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The effect of small additions of niobium upon the corrosion rates and electrochemical potentials of iron in HCl, HF, and H_2SO_4

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Department:

THE EFFECT OF SMALL ADDITIONS OF NIOBIUM UPON THE CORROSION RATES AND ELECTROCHEMICAL POTENTIALS OF IRON IN HCl, HF, AND H_2SO_4

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THESIS

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ABSTRACT

The valence of niobium ions going into solution was determined by dissolving the metal in hot HF and measuring the volume of hydrogen evolved.

The corrosion rates of Nb, NbH_x, Fe and Fe - Nb alloys were determined in various concentrations of HCl, H_2SO_4 and HF. The Fe and Fe - Nb alloy samples were made by using powder metallurgy techniques.

It was found that a metal skin was produced on the surface of iron corrosion samples if the carbon content of the surface layer was lower than that of the core of the sample. The lower carbon content of the surface was caused by sintering in a H_2 atmosphere.

The electrochemical potentials of Nb, NbH_x, Fe and Fe - Nb alloys were determined in the same acids in which the corrosion studies were performed.

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I. INTRODUCTION

Niobium was first discovered in 1801 in columbite by The Englishman Hatchett. He named it columbium after Columbia, "Gem of the Ocean". In 1844 niobium was rediscovered by H. Rose who found it in close association with tantalum in columbite. Rose named what he thought was a new metal niobium after the goddess Niobe, daughter of Tantalus. The dual identity of the metal has led to much confusion and pages of discussion in the scientific literature. The American Chemical Society agreed in an international conference to call it niobium and we will call it niobium throughout the rest of this paper, as proposed by the Society, although the metallurgists (U.S.A.) are still calling it "columbium".

Before 1957 the main use of niobium was in the form of ferroniobium as an addition to stainless steels and high-temperature alloys of the nickel and cobalt-base types to form stable carbides. The metal itself has such a limited use that only a few tons have been produced since its discovery. The nuclear field was the first to show interest in niobium as an engineering material. It offered the advantages of low neutron-capture cross section and high-temperature strength as a cladding material for fuel elements in nuclear reactors. Niobium was also found to be ductile and to have good cold workability so that it can be rolled into sheet and rod,

stamped, and drawn into wire and tubing. It can also be readily machined and welded. The melting point of niobium is 2468°C [1], which is not far below that of molybdenium, placing it among the refractory metals. Although niobium is not as light as aluminum or magnesium, it is much lighter than nickel or cobalt and over 15% lighter than molybdenum. Its density is 8.4 g cm^{-3}; that of Mo, Ni, and Co are 10.2 g cm^{-3}, 8.9 g cm^{-3} and 8.9 g cm^{-3} respectively. These properties have broadened the fields of application of niobium to include use at high temperatures in gas turbines, ram jets, airframes, and missiles.

The resistance of niobium to weathering is excellent, and general corrosion resistance is considered to be very good. The object of this thesis was to investigate the effect of small alloying additions of niobium to iron on the latter's corrosion rate. To better understand the corrosion processes involved, the effect of the same alloying additions on the electrochemical potential of iron in acid media was also studied. However, since powder metallurgy methods were used for the preparation of the samples, some knowledge of the corrosion behavior of such iron samples was also necessary. Finally, since

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[1] T. H. Schofield, "The Melting Point of Niobium," J. Inst. of Metals, LXXXV, April (1957), 372.

very little is known about the corrosion resistivity of niobium itself, this metal, in the forms of powder and sheet, was studied first.

II. LITERATURE SURVEY

Since 1948 there have been several similar articles published which deal with the corrosion properties of niobium. Perhaps the earliest of these is by C. W. Balke[2] in the Corrosion Handbook. He states that niobium is highly resistant to corrosion only at ordinary temperatures. An increase of temperature to 100°C results in a greatly increased corrosion rate. Niobium is much more active than tantalum and much more susceptible to hydrogen embrittlement. Hydroflouric acid, or a mixture of HF and HNO_3, dissolves the metal completely, and in some media, even where the weight loss is small, the metal becomes very brittle.

Taylor[3], Fontana[4], Inglis and Cotton[5], Badger[6],

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[2] C. W. Balke, The Corrosion Handbook, ed. H. H. Uhlig, (New York: John Wiley and Sons, 1948), p. 60.

[3] Donald F. Taylor, "Acid Corrosion Resistance of Tantalum, Columbium, Zirconium, and Titanium," Industrial and Engineering Chemistry, April, 1950, 639.

[4] Mars G. Fontana, "Corrosion Resistance of Tantalum, Niobium, Zirconium, Tungsten, and Molybdenum," Industrial and Engineering Chemistry, July, 1952, 71A.

[5] J. B. Cotton and N. P. Inglis, "Corrosion Behavior of Some of the Newer Metals," Corrosion Prevention and Control, V, No. 11, (1958), pp. 59 - 73.

[6] F. S. Badger, "High Performance Materials, Alloys and Reactive Metals," Industrial and Engineering Chemistry, L, No. 11, (1958), 1608.

Hampel[7], Macleary[8], Stuckens and Durand[9], Buchman[10], and Edwards[11] all report observations very similar to Balke's which are summarized as in Table I.

No literature was located which deals with the corrosion of pure iron produced by powder metallurgy. The only literature found concerning the corrosion of iron containing niobium was about steel with niobium additions. The niobium takes different forms in these two systems due to the fact that in steel the niobium is tied up as a carbide. Therefore, the effect of niobium additions on the corrosion of steel cannot be extended to iron.

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[7] Clifford A. Hampel, "Corrosion Properties of Tantalum, Columbium, Molybdenum, and Tungsten," Corrosion, XIV, No. 12, (1958), 29.

[8] Donald L. Macleary, "Testing of Columbium and Columbium Alloys," Corrosion, XVIII, (1962), pp. 67t - 69t.

[9] W. Stuckens and R. Durand, "Corrosion of Niobium as a Function of Atmosphere and Temperature," Corrosion Anticorrosion, XI, No. 5, (1963), pp. 180 - 189.

[10] W. T. Buchmann, "Don't Overlook Refractory Metals for Corrosive Environments," Materials in Design Engineering, LXIV, No. 6, (November, 1966), pp. 106 - 108.

[11] W. T. Edwards, Corrosion, ed. L. L. Shreir (New York: John Wiley and Sons, 1963), pp. 5.19 - 5.31.

TABLE I

SUMMARY OF PUBLISHED DATA ON THE CORROSION OF NIOBIUM

CORRODANT	TEMPERATURE °C	TEST PERIOD DAYS	CORROSION RATE MM/YEAR
HCl, 20%	19-26	82	0.00000
HCl, Concd.	19-26	82	0.00254
HCl, Concd.	110	67	0.1016
HNO_3, Concd.	19-26	67	0.00000
HNO_3, Concd.	100	67	0.00000
1 HNO_3, 2 HCl	19-26	35	0.00051
1 HNO_3, 2 HCl	50-60	1	0.0254
H_2SO_4, 20%	19-26	3650	0.00000
H_2SO_4, 20%	95-100	4	0.00051
H_2SO_4, 25%	19-26	3650	0.00000
H_2SO_4, 40%	19-26	3650	0.00000
H_2SO_4, 50%	19-26	3650	0.00000
H_2SO_4, Concd.	19-26	3650	0.00254
H_2SO_4, Concd.	50	67	0.02032
H_2SO_4, Concd.	95-100	32	0.4826
H_2SO_4, Concd.	145	30	0.4572
H_2SO_4, Concd.	175	1	36.068+
H_2SO_4, Concd. + CrO_3	100	42	0.2032
H_3PO_4, 85%	19-26	82	0.00000
H_3PO_4, 85%	100	31	0.08382

TABLE I (continued)

HF, ALL	*19-26*	*1*	*Very High*
$FeCl_3$, 10%	*19-26*	*36*	*0.00000*
Oxalic Acid, 10%	*19-26*	*82*	*0.01524*
Oxalic Acid, 10%	*100*	*82*	*0.01524*
NH_4OH	*19-26*	*82*	*0.00000*
Na_4CO_3, 20%	*100*	*50*	*0.03302*
NaOH, 5%	*19-26*	*31*	*0.02794*
NaOH, 5%	*100*	*5*	*0.4826*
KOH, 5%	*19-26*	*31*	*0.2032*
KOH, 5%	*100*	*5*	*1.1738*
M_2O_2, 30%	*19-26*	*61*	*0.00508*

III. EXPERIMENTAL

A. MATERIALS

1. Niobium Powder

The niobium powder used in this investigation was obtained from the Battele Memorial Institute, which received it from H. C. Starck AG, a German firm. It was produced by the reduction of K_2NbF_7 with sodium vapor. The analysis which appears in Table II shows the purity of the metal comparable to or better than most other commercial grades of Nb powder. The high value of oxygen associated with the powder, according to the description of the preparation, is not exactly in solid solution with the metal, but mostly on the particle surfaces as a Nb oxide and as adsorbed oxygen. H_2O may also be adsorbed on the surface of the particles. With proper sintering, it is believed, the content of oxygen can be reduced substantially.[12]

2. Niobium Sheet

The niobium sheet used in this investigation was also obtained from the Battele Memorial Institute. It was fabricated from electron-beam ingot stock previously

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[12] W. D. Klopp, C. T. Sims, and R. I. Jaffe, "Vacuum Reactions of Niobium During Sintering," Technology of Columbium, ed. B. W. Gonser and E. M. Sherwood (New York: John Wiley and Sons, Inc., 1958), pp. 106 - 120.

TABLE II

CHEMICAL ANALYSIS OF NIOBIUM POWDER IN PARTS PER MILLION AND PERCENT.

ELEMENT	*PPM*	*PERCENT*
Oxygen	*6000*	*0.6*
Hydrogen	*30*	*0.003*
Nitrogen	*300*	*0.03*
Carbon	*300*	*0.03*
Tantalum	*1200*	*0.12*
Niobium by difference	*----*	*99.217*

obtained from the Stauffer-Temescal Metals Company. The sheet had been reduced in thickness more than 99%, having been cold worked at room temperature. It had a Vicker's hardness of 165. The analysis of the sheet is given in Table III. This is probably the highest purity niobium available from any present day commercial suppliers of the metal.

3. Iron Powder

The iron powder used in the present investigation was obtained from the Fischer Scientific Company. The powder was produced electrolytically and its analysis appears in Table IV, as supplied by the company.

4. Acid Solutions

The acids used, HCl, H_2SO_4, and HF, were of reagent grade and conformed to ACS standards. The solutions were prepared by dilution of the concentrated acids with distilled water and standardized by titration with freshly dried and weighed amounts of Na_2CO_3, using methyl orange as an indicator.

B. DETERMINATION OF THE VALENCE OF NIOBIUM DISSOLVING IN HF

1. Apparatus and Procedure

The apparatus used for this determination is shown in Figure 1. It consists of a 100 ml. gas burette with divisions every 0.2 ml., a leveling bulb to maintain

TABLE III

CHEMICAL ANALYSIS OF NIOBIUM SHEET IN PARTS PER MILLION.

ELEMENT	PPM
Oxygen	100
Hydrogen	1
Nitrogen	40
Carbon	40
Tantalum	1000
Calcium	10
Silicon	N.D.
Zirconium	30
Copper	N.D.
Chromium	N.D.
Iron	N.D.
Magnesium	N.D.
Molybdenum	30
Nickel	N.D.
Titanium	30

N.D. means not detected

TABLE IV

CHEMICAL ANALYSIS OF ELECTROLYTIC IRON POWDER IN PERCENT BY WEIGHT

ELEMENT	PERCENT
Total Iron	98.060
Metallic Iron	93.500
Oxygen	1.850
Carbon	.015
Manganese	.007
Phosphorus	.004
Silicon	.007
Sulfur	.008
Copper	.010
Chromium	.002
Nickel	.015
Molybdenum	.002
Vanadium	.003
Cobalt	.007
Tin	.005
Aluminum	.002
Mercury	nil

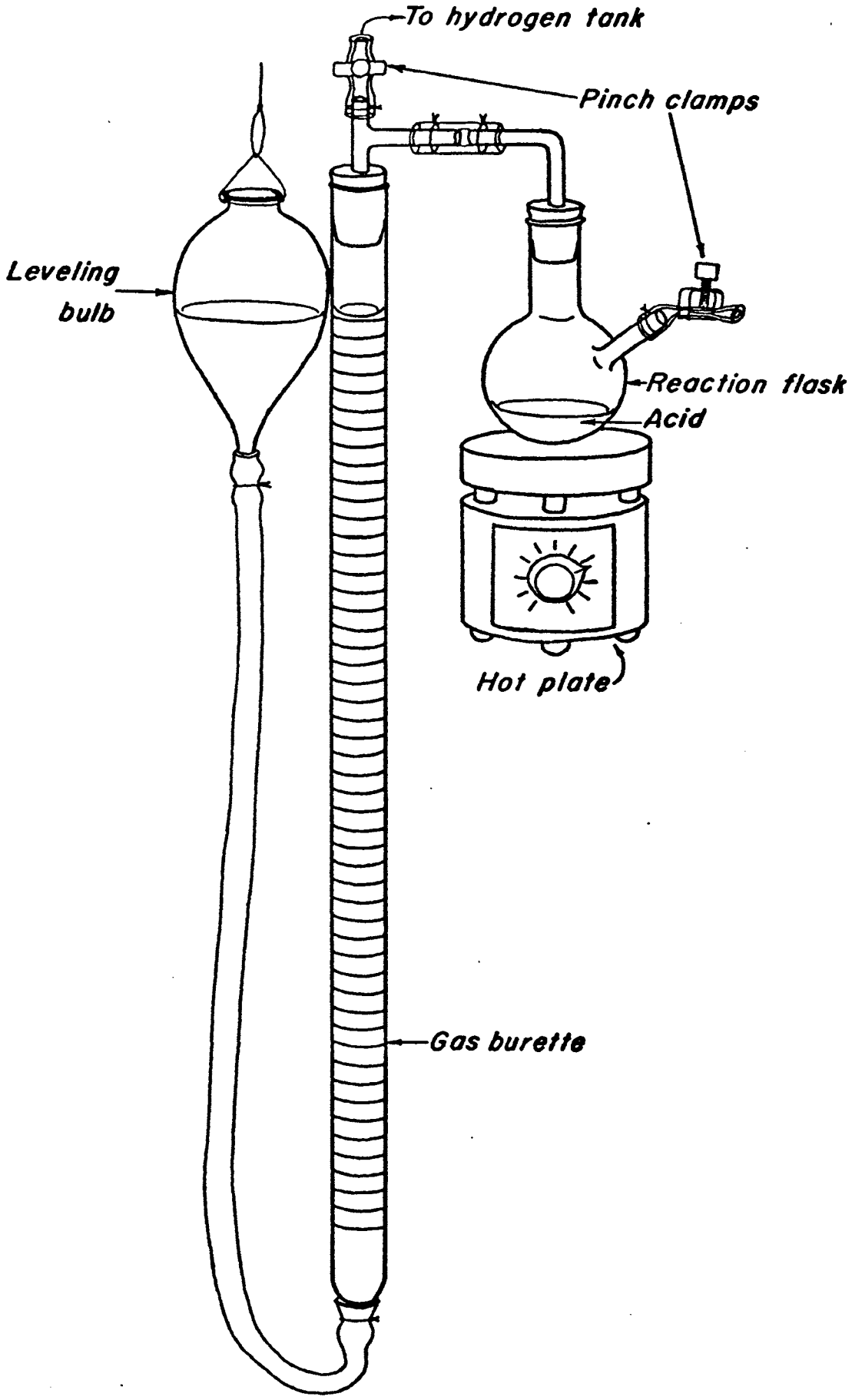

FIGURE 1. APPARATUS FOR THE DETERMINATION OF THE VALENCE OF NIOBIUM IONS FORMED IN HOT HF.

atmospheric pressure in the burette, a small 50 ml. reaction flask, a small hot plate, and glass and rubber tubing to complete the apparatus as shown.

Niobium has only negligible dissolution rates in most acids, but in heated HF it dissolves readily giving off hydrogen. Its volume is proportional to the valence of the metal ions formed, which is a small whole number (1, 2, 3, 4, etc.). Every gram equivalent of a dissolving active metal corresponds to 11,207 ml. of hydrogen. Generally, the

$$\text{atomic mass} = (\text{valence number})(\text{equivalent mass}) \qquad (1)$$

and the equivalent mass is determined from the volume of H_2 in ml. developed by X g. of metal.

$$\text{equivalent mass} = X(11{,}207)/\text{volume of } H_2 \text{ in ml.} \qquad (2)$$

Since according to (1)

$$\text{valence number} = \text{atomic mass}/\text{equivalent mass}$$

then, using (1) and (2) we get

$$\text{valence number} = (\text{atomic mass})(\text{volume } H_2 \text{ dev.})/X(11{,}207) \qquad (3)$$

and for niobium specifically,

$$\text{valence} = 92.906(\text{volume } H_2 \text{ developed})/X(11{,}207)$$

Hence, the valence can be found from a fairly simple equation (3), adjusted for niobium (at. mass = 92.906).

To obtain the values to put into this equation the reaction flask of Figure 1 was first dried in an oven at 250°C over night, then cooled in a desiccator and weighed. While the flask was still on the semi-micro balance a small amount of Nb powder was put into it. The reaction flask was then removed from the balance and about 25 ml. of cold 5N HF was poured in, being careful not to splash out any Nb powder. The dissolution rate of the niobium in cold 5N HF was so small that no hydrogen bubbles appeared and if any hydrogen was lost in the time before sealing the system, it was negligible.

The flask was then tightly connected to the crossover tube by means of a rubber stopper. A large beaker of water was then put under the flask so that the water level reached the base of the neck of the flask. A thermometer was inserted into the water and the system was purged with hydrogen for at least fifteen minutes and then tightly closed by means of the pinch clamps. The level of the water in the leveling bulb was matched to that in the burette which was usually slightly below zero. This was because the system was closed with a little pressure still inside to prevent any air from entering. Then the temperature of the bath and reaction flask was given about five minutes to stabilize and then

the temperature was recorded along with that of the room and the water level in the burette. The beaker of water was then removed and a small hot plate was placed under the reaction flask about 2 mm. from its bottom (see Figure 1). The temperature of the hot plate was increased slowly until the niobium started to dissolve, as evidenced by the appearance of H_2 bubbles on the surface of the powder. The temperature never exceeded the boiling point of the acid. The end of the reaction was recognized by ceasing of hydrogen evolution. This usually took 45 to 60 minutes for each milligram of niobium. However, the powder did not always dissolve completely. Throughout the reaction time the water level in the leveling bulb was kept even with that in the burette to avoid any pressure within the system.

Upon ceasing of the hydrogen evolution the hot plate was replaced by the water bath in the same position as before the dissolution. The temperature of the bath was adjusted exactly to that at the beginning of the experiment, and allowed to stabilize for about ten minutes. Then, the volume of gas was recorded, making sure that it was read on a horizontal plane. The room temperature was also recorded along with the barometric pressure. The reaction flask was then removed from the system, and the acid diluted with distilled water being careful not to disturb the residue

on the bottom of the flask. The diluted acid was then sucked out of the flask leaving the residue. The residue was again rinsed gently but thoroughly with distilled water and then the flask was put into an oven to dry over night. The flask was cooled in a desiccator and then weighed. The niobium residue was then carefully removed and the flask was weighed again, the difference in weights being the weight of the residue. To calculate the valence of niobium using equation (3), it was necessary to reduce the volume of the gas to standard temperature, pressure, and dryness. In recording the pressure, brass scale and capillary depression corrections were also made. Sample calculations appear in the Appendix.

The most critical features of the technique described above are: (1) that the system is well sealed so that no hydrogen escapes, (2) that the system is well purged with hydrogen to prevent any hydrogen being lost due to H_2O formation with oxygen from the air, and (3) that the temperature is kept only high enough for the niobium to dissolve with no boiling and a minimum of water vapor condensation in the crossover tube.

2. Results and Conclusions

All the data are shown in the Appendix on pages 78 through 81 . The experiment was run four times to increase the probability of the average. Table V

on the next page summarizes the results showing the amount of niobium dissolved, the corrected volume of hydrogen collected, and the calculated valence of niobium.

The results on the next page show that niobium dissolves in heated HF with a valence of plus five according to the following equation:

$$Nb = Nb^{+5} + 5e^{-} \qquad (4)$$

or,

$$Nb + 5H^{+} = Nb^{+5} + 2.5H_2 \qquad (4a)$$

C. CORROSION RATES OF NIOBIUM, NIOBIUM HYDRIDE, IRON, AND IRON-NIOBIUM ALLOYS

1. Apparatus and Procedure

The iron samples used for the corrosion rate determinations were made by putting about 15 grams of the Fe powder into a small tube furnace and treating the powder at 600°C for three hours in hydrogen. After heating, the powder was furnace cooled, under the gas. By this step, part of the carbon impurity in the iron was extracted and the oxide reduced. With this treatment the skin formation discussed below was prevented. The iron powder was removed from the furnace and ground with a mortar and pestle to separate the particles of the partially sintered powder.

TABLE V

SUMMARY OF NIOBIUM VALENCE NUMBER DETERMINATION DATA

RUN NUMBER	WEIGHT Nb DISSOLVED	CORRECTED H_2 VOLUME	CALCULATED VALENCE OF Nb
1	.00499 g.	3.04 ml.	5.050
2	.00867 g.	5.14 ml.	4.915
3	.00873 g.	5.22 ml.	5.004
4	.00614 g.	3.72 ml.	5.023
		Average	4.998

Then 2.5 g. of the iron sample were weighed, put into a die and pressed into a pellet under 56,000 psi. The pellets were then sintered in a high temperature furnace at 1350°C for two hours in a hydrogen atmosphere, and furnace cooled. The furnace used for sintering was a Cambridge Materials Research High Temperature Laboratory Furnace Type PCA10 obtained from England. The furnace is shown in Figure 2 with the crucibles, alumina spacers and iron samples in place.

After removal of the samples from the furnace a 1.5 mm. diameter hole was drilled near the edge of each sample so that they could be hung into the acid as shown in Figure 3. This hole was drilled far enough from the edge so that there was little chance of breaking through during corrosion.

The iron-niobium alloy samples were made essentially in the same way. The difference was that the right percentage of niobium and of hydrogen treated iron had to be weighed out on an analytical balance to make 2.5 g. Thorough mixing was necessary in a clean mortar with a pestle to assure that all the samples would be uniform.

In the production of the iron and iron-niobium alloy samples the objective was to obtain these samples without preparing the molten metals. Powder metallurgy methods render this possible, and sintering allows the particles to fuse together without actually melting.

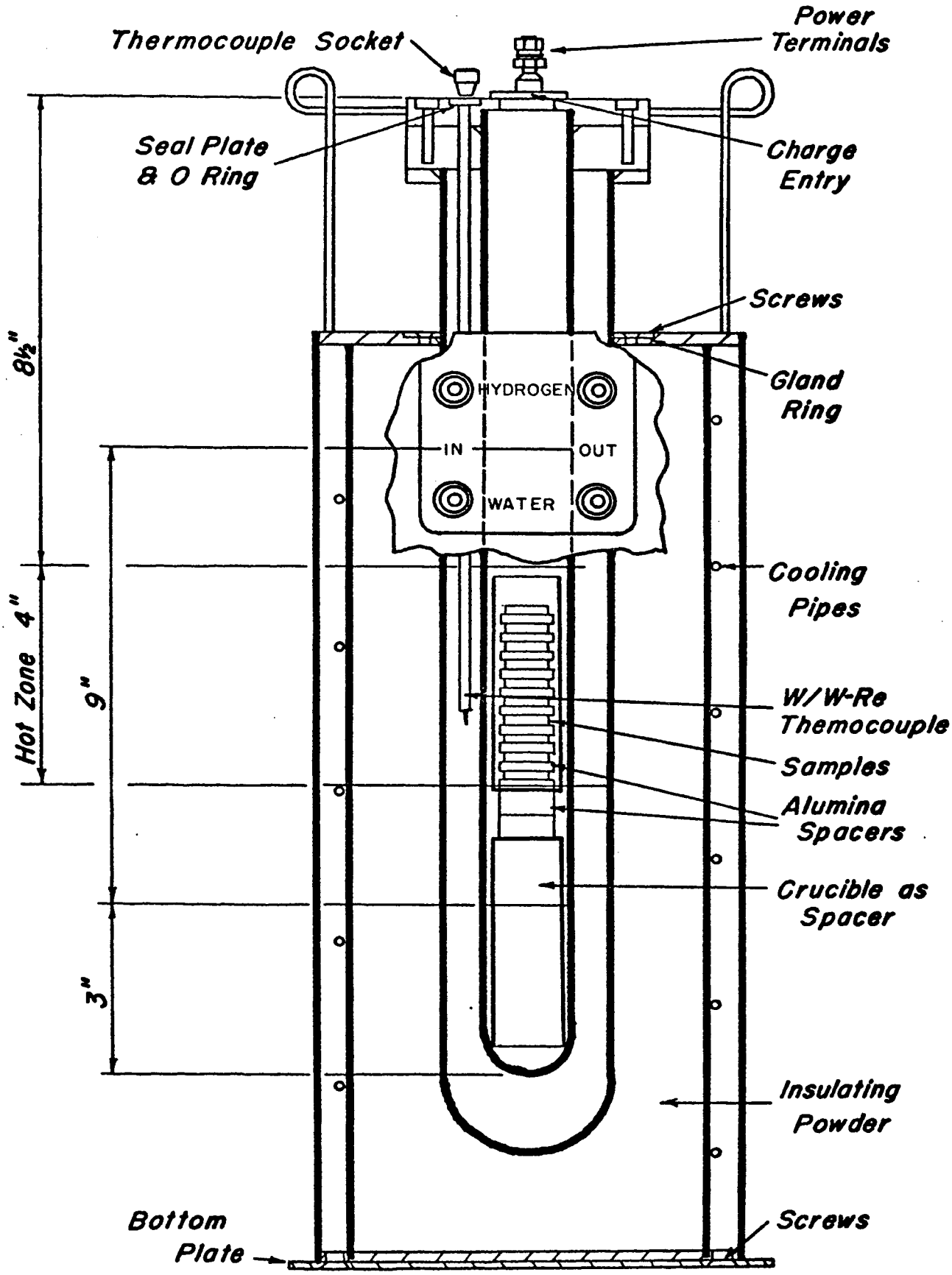

FIGURE 2. MATERIALS RESEARCH LTD. HIGH TEMPERATURE LABORATORY FURNACE TYPE PCA10 (UP TO 1800°C) USED TO SINTER IRON AND IRON-NIOBIUM ALLOY SAMPLES.

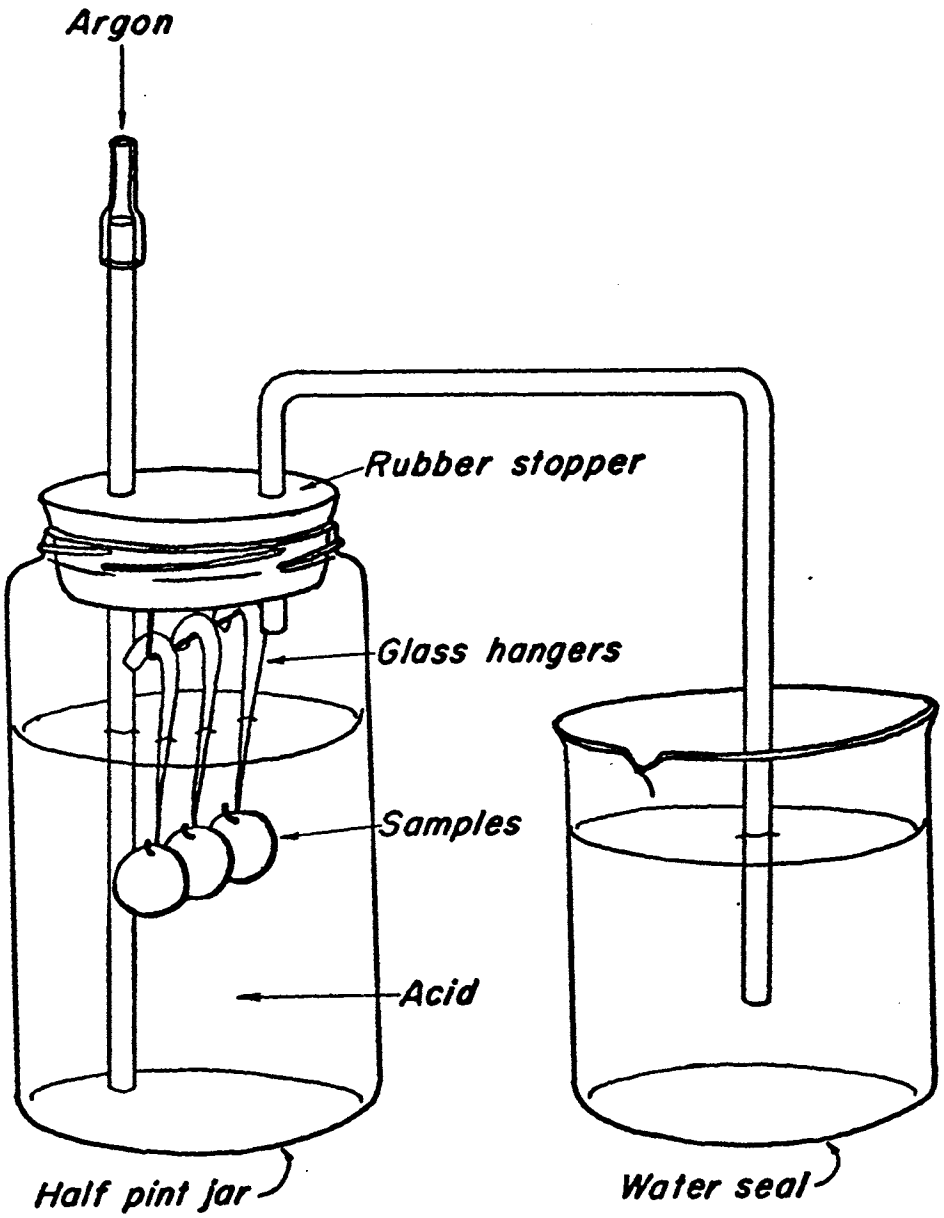

FIGURE 3. APPARATUS FOR THE DETERMINATION OF THE CORROSION RATES OF SAMPLES WITHOUT THE PRESENCE OF OXYGEN.

Sintering also permits the different metals mutually to diffuse as in a molten alloy, only more slowly.

The highest practical temperature possible was chosen because the mechanisms which bring about interdiffusion and reduce concentration gradients are temperature dependent.

From Figure 4 it can easily be seen that 1350°C is a temperature high enough to sinter the niobium-iron alloys without the danger of melting.

Two other influential factors in sintering are the sintering atmosphere and the pressure applied to the compact. Sintering atmospheres can promote or inhibit the sintering process. Aggressive or nascent gases usually promote sintering of metals. Sintering atmospheres must also be kept as free of oxygen and moisture as possible. The absorption of oxygen affects mass transport in iron by simultaneously lowering the driving force and impeding surface migration.[13] *Hydrogen atmospheres have been reported*[14] *to promote sintering in iron powder metallurgy with good results in regard to mechanical properties and density. For these reasons dry hydrogen was chosen as the sintering*

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[13] *F. Thummler and W. Thomma, "Metallurgical Review No. 115: The Sintering Process," Metals and Materials, I, No. 6, (June, 1967), 90.*

[14] *Ibid., p. 91.*

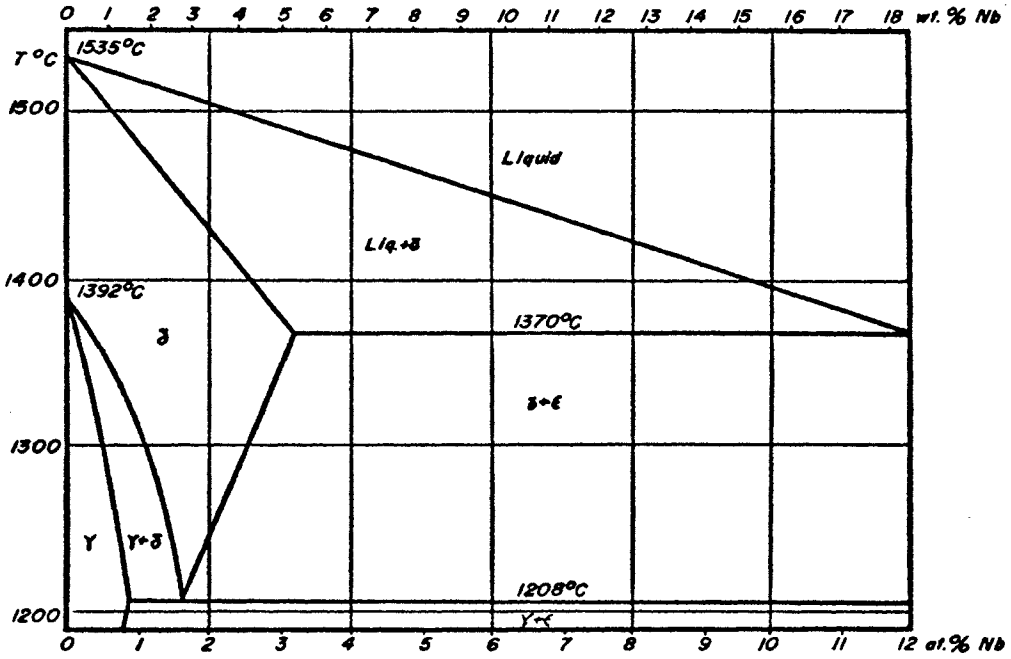

*FIGURE 4. HIGH TEMPERATURE, IRON RICH SIDE OF NIOBIUM-IRON PHASE DIAGRAM.**

* *A. Ferrier, E. Ubelacker, and E. Wachtel, "Nb-Fe Phase Diagram from 0 to 12% Nb and 1200 to 1535°C," Comptes Rendus, CCLVIII, No. 22, (June, 1964), 5426.*

atmosphere in this investigation.

Pressing influences sintering in three ways: (a) it raises the contact area per unit volume, thus producing an increase in unsintered density, (b) it introduces elastic and plastic deformation, and (c) it deforms and cracks surface layers. However, pressing furthers the inclusion of air and gases. In general, the higher the compacting pressure the more the sintering process is promoted. But such a behavior is by no means true in every case. The value of 56,000 psi. was chosen for this investigation because it was the highest pressure available.

There are three distinguishable stages of isothermal sintering which merge continuously into one another.[15] *The first stage is the growth of a neck between particles which touch each other. This process proceeds according to an exponential time law.*[16] *During this stage the particles remain as individuals with no strong grain growth beyond the original particle.*

The second stage is that of densification and grain growth in which neck growth is intensified and the separate particles begin to lose their identity. Most of the shrinkage takes place during this stage and a

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[15] *Ibid.*, p. 71.

[16] *Ibid.*

network of pores is formed. When about 90% of the theoretical density is reached the proportion of closed pore volume increases rapidly, leading to the final stage of sintering.

In the final stage nearly all of the pores are closed. The isolated pores become increasingly spheroidized and further densification is so slow that it is very difficult, except by density measurements, to determine when the process is at its end.

There are several possible growth mechanisms which can occur during sintering of particles touching each other. At first they may adhere, though this is of little importance in the sintering process employed here. Recovery and recrystallization alone are incapable of producing sintering, however they are responsible for the grain growth which accompanies densification. Material transport by vaporization and re-condensation is very unlikely in the present case because this process cannot produce shrinkage which was observed with the samples. It can be assumed that surface diffusion occurs in nearly every sintering process. It causes the rounding-off of all the particle edges, corners, and pores and starts at comparatively low temperatures due to the low activation energy of the process.[17]

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[17] *Ibid.*, p. 73.

Grain-boundary diffusion is often inferred from calculated activation energies for shrinkage, although it alone cannot explain the contractions observed. Interstitial diffusion with respect to sintering has been studied very little, but could be an important mechanism in solid solution formation.

The most significant mechanism seems to be lattice diffusion by way of vacancy migration. The diffusion process during sintering is governed by the type and geometrical arrangement of the vacancy sources and sinks. Vacancy sources may be small pores, concave surfaces, and dislocations. Sinks are grain boundaries, plane or convex surfaces, large pores, as well as dislocations.

There are many factors which influence such a material transport. They fall into three general catagories: (a) temperature-dependent properties of the material (including structure and state of bonding), (b) powder properties, such as oxide layers on the particles, pre-treatment along with sintering conditions, and (c) foreign constituents (including those introduced by the sintering conditions).

Sintering of a multicomponent system, such as iron-niobium, introduces several factors which influence sintering. The most important of these are the extent of mutual solubility and the possibilities of the formation of new phases or of a solid solution. It is

clear from Figure 5 that, indeed, a new phase should appear in samples low in niobium. According to Figure 5 (right side) samples containing up to 5% Nb by weight should, at room temperature, consist of a mixture of $NbFe_2$ and ferrite. The $NbFe_2$ should be uniformly distributed throughout the samples if proper sintering conditions are observed.

Figure 6 shows the microstructure of a pure iron sample and Figures 7 - 12 show the microstructure of increasing amounts of Nb in the iron. The alloy containing .5% by weight of Nb is represented by Figure 7 and the $NbFe_2$ phase can be seen in small pockets along the grain boundaries. Very similar is Figure 8 showing an alloy with 1% Nb by weight, but with more of the $NbFe_2$ in the grain boundaries. Figure 9 (2% Nb by weight) shows many small grains of the $NbFe_2$ distributed through the ferrite matrix and not just in the grain boundaries. Figures 10, 11, and 12 (3%, 4% and 5% by weight Nb respectively) show increasing amounts of $NbFe_2$ and the development of what appears to be a lamellar eutectic type structure. Figure 12 depicts an additional formation. It appears as though the $NbFe_2$ formed larger grains of its own which are lined up in what may have been the original ferrite grain boundaries. All of these figures are representative of their respective samples and they show that the Nb was

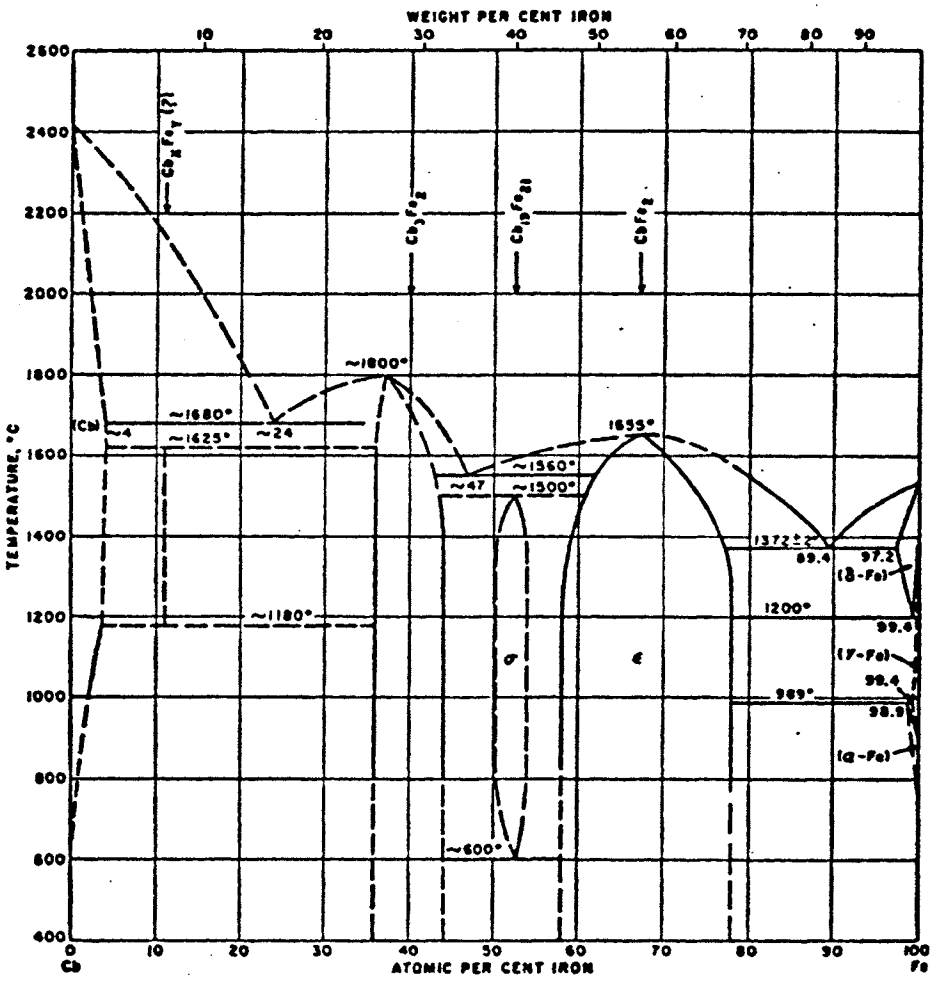

*FIGURE 5. THE Nb-Fe PHASE DIAGRAM.**

* R. P. Elliott, Constitution of Binary Alloys, First Supplement (New York: McGraw-Hill Book Co., 1965), 255.

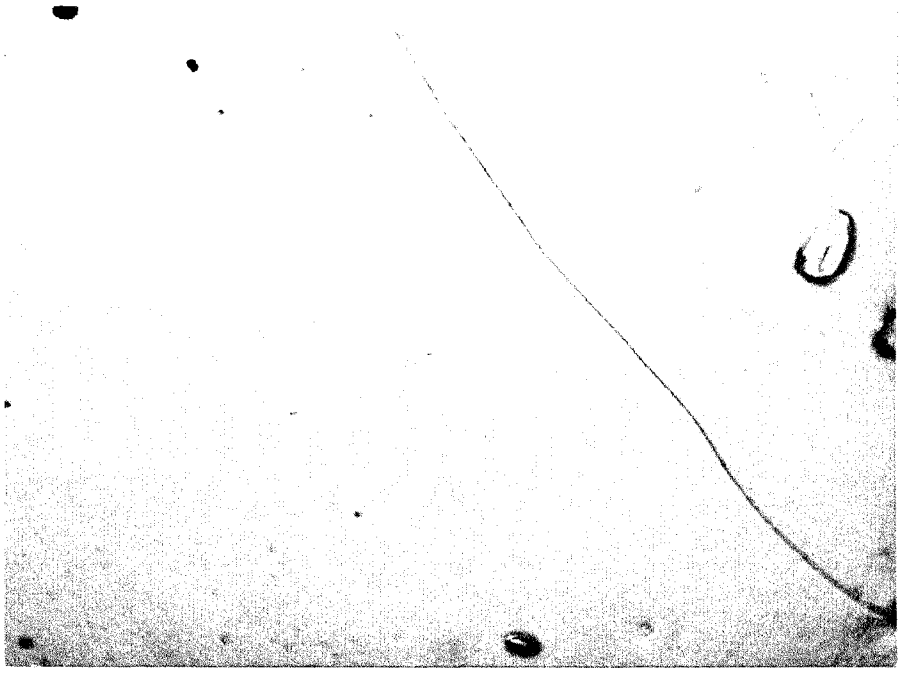

FIGURE 6

Microstructure of electrolytic pure iron powder pressed at 56,000 psi and sintered at 1350°C two hours under dry hydrogen. (500X-2% nital etch)

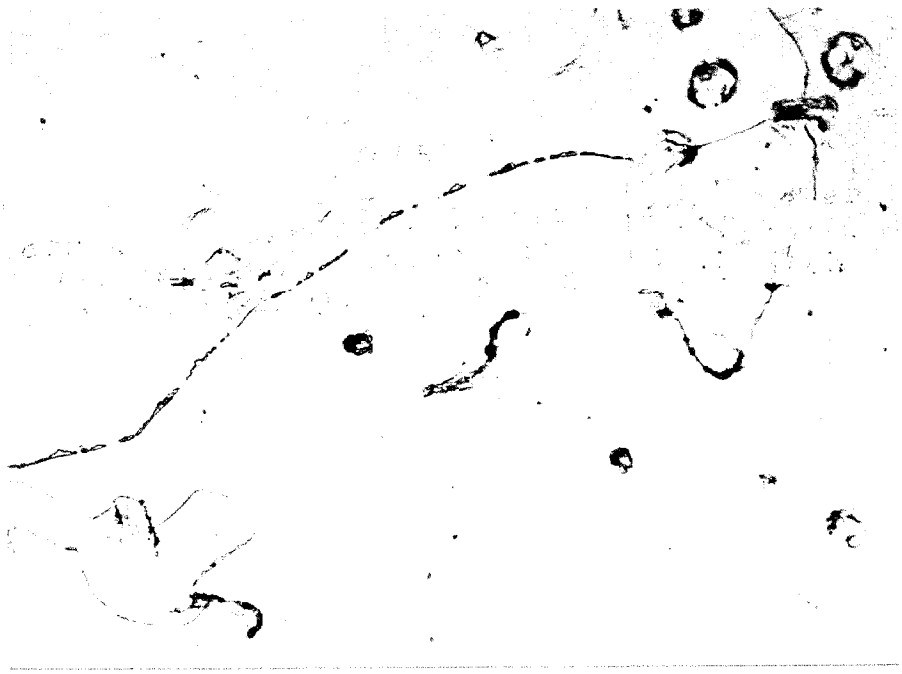

FIGURE 7

Microstructure of electrolytic pure iron powder plus .5% niobium powder prepared as in Figure 6 . (500X-2% nital etch)

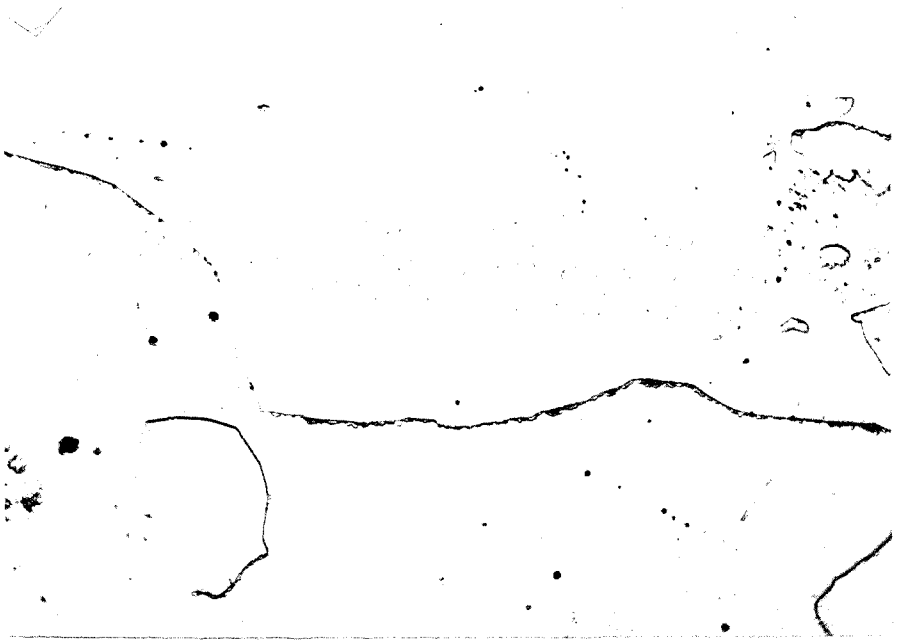

FIGURE 8

Microstructure of electrolytic pure iron powder plus 1% niobium powder prepared as in Figure 6 . (500X-2% nital etch)

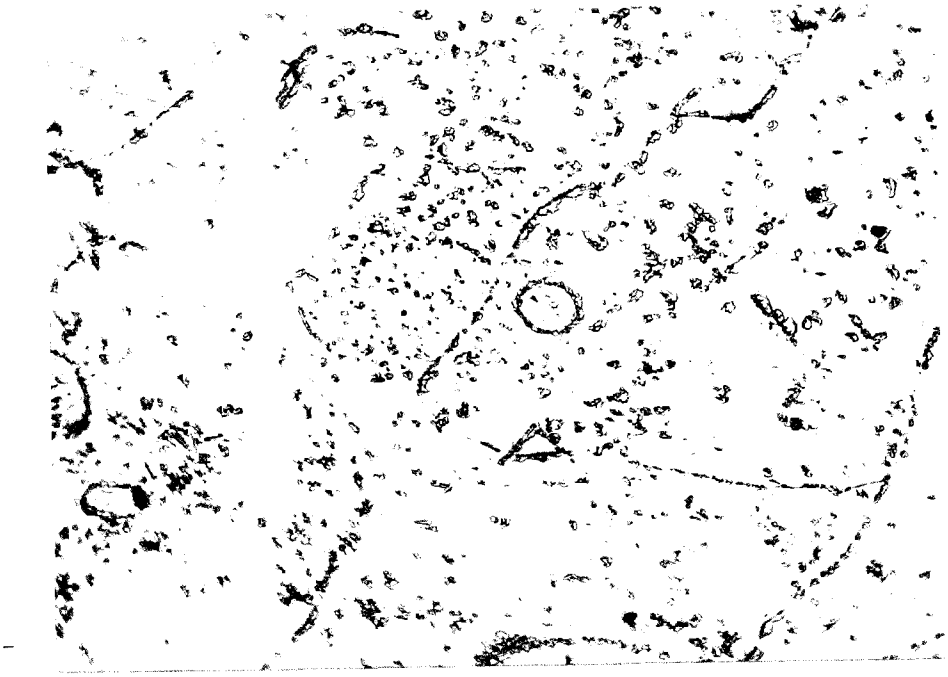

FIGURE 9

Microstructure of electrolytic pure iron powder plus 2% niobium powder prepared as in Figure 6 . (500X-2% nital etch)

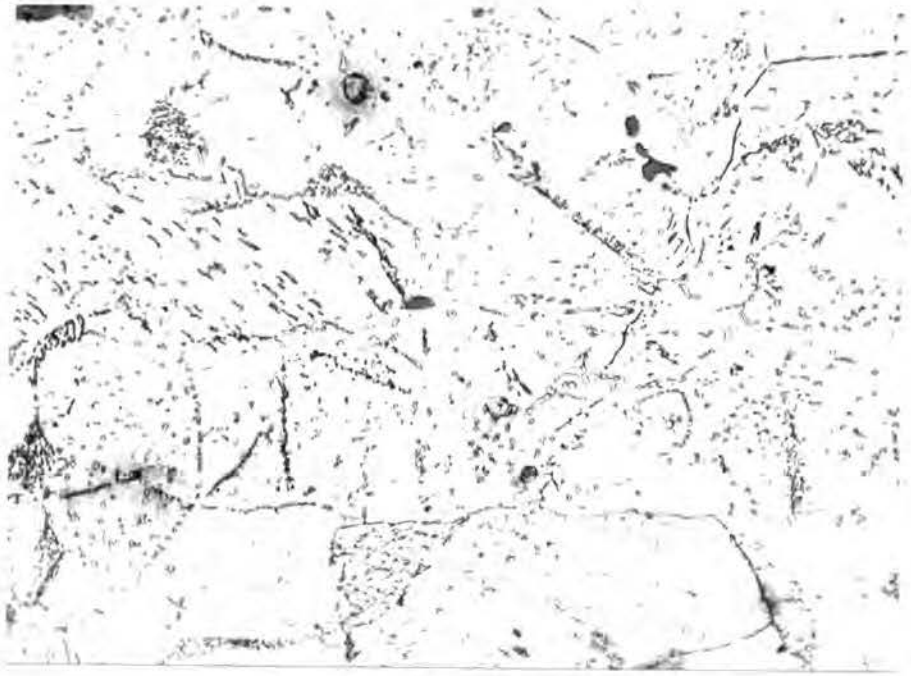

FIGURE 10

Microstructure of electrolytic pure iron powder plus 3% niobium powder prepared as in Figure 6 . (500X-2% nital etch)

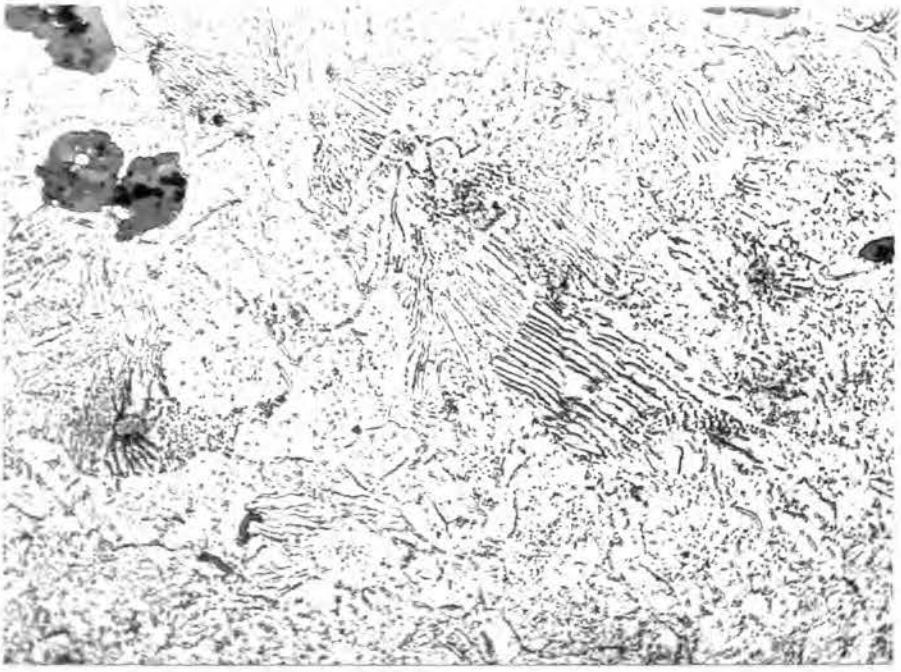

FIGURE 11

Microstructure of electrolytic pure iron powder plus 4% niobium powder prepared as in Figure 6 . (500X-2% nital etch)

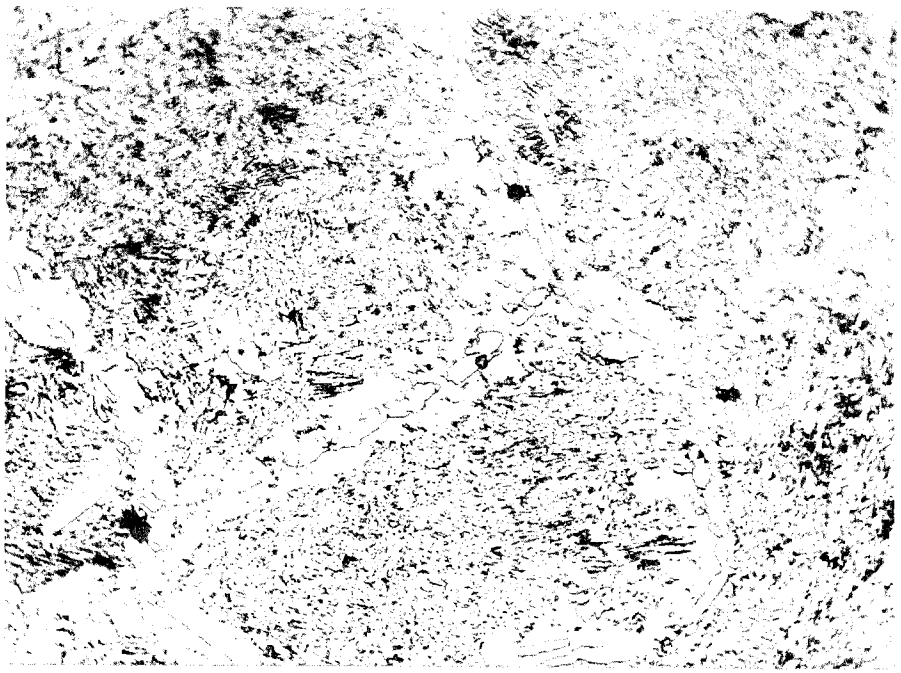

FIGURE 12

Microstructure of electrolytic pure iron powder plus 5% niobium powder prepared as in Figure 6. (500X-2% nital etch)

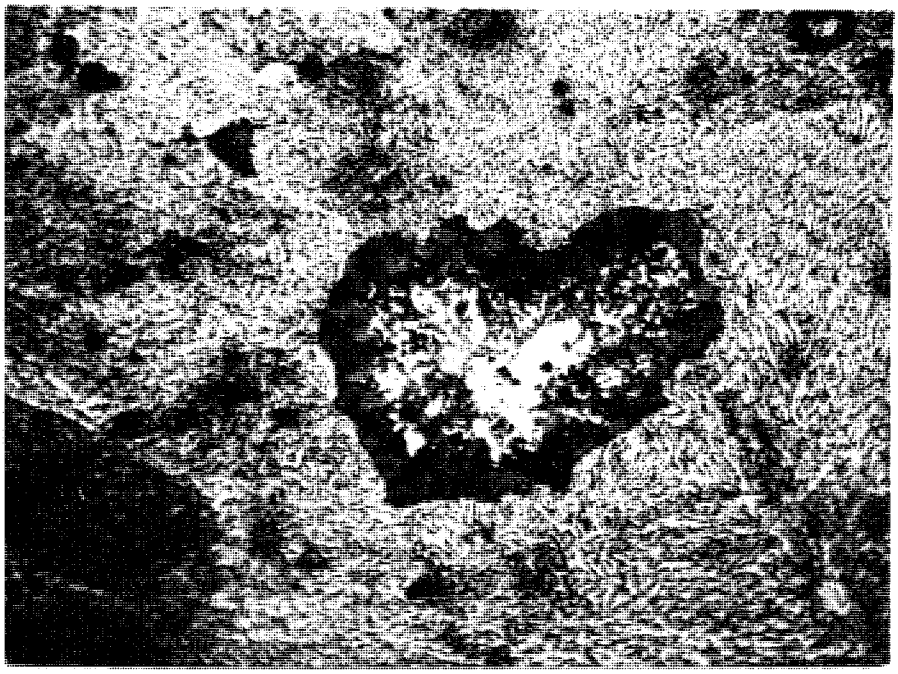

FIGURE 13

Microstructure of unusual oxide particles found in iron-niobium alloy samples. This one is in 96%Fe-4%Nb. (500X-2% nital etch)

thoroughly distributed during sintering.

Figure 13 is included here to show the microstructure of unusual oxide particles which were found in the iron-niobium alloy samples. Evidently the sintering time was insufficient to reduce all the oxide. It appears that the oxide is surrounding some metallic phase in its core. This phase was determined, upon examination with the electron microprobe, to be niobium with a small amount of iron.

The niobium samples used in this investigation were cut from a niobium sheet in squares about one centimeter on each edge. Holes 1.5 mm. in diameter were drilled near one corner to facilitate hanging of the samples in the acid, as shown in Figure 3. It was initially decided to make the niobium samples, as with the iron samples, by pressing the powder and sintering it. This was not possible due to the lack of a furnace with a temperature range high enough to adequately sinter the niobium in an inert atmosphere.

The niobium hydride samples were prepared by first cutting squares from niobium sheet, drilling holes in them as described, and placing them into a tube furnace. The niobium squares were then heated in a hydrogen atmosphere at 900°C for three hours and then cooled, still under hydrogen.

After all the samples had been made as described above they were ground with 600 grit paper on all surfaces. All dimensions necessary to calculate the surface areas of the samples were measured with a vernier caliper and, finally, the samples were thoroughly washed with distilled water and acetone to remove fingerprints and dirt. Then the samples were carefully weighed on an analytical balance and their weights were recorded.

The apparatus used to corrode the samples is shown in Figure 3. Argon was bubbled through the acid fairly quickly at first to purge out all oxygen. After about ten minutes the rate of flow of argon was reduced to about one bubble every five seconds.

Once a day, at the same time every day, the samples were removed from the acid, rinsed well with distilled water, washed with acetone, and dried. They were then weighed and returned to the acid for another 24 hour period. The difference in weight from one day to the next was converted to corrosion rate or velocity (v) as shown in equation (5),

$$v = w/At \qquad (5)$$

where w is the weight loss in milligrams (mg), A is the surface area in cm^2 and t is the time in hours. Thus the corrosion rate is given in mg/cm^2hr. Three samples of the same origin were usually placed into

the same bottle of acid and the rates obtained were averaged to get the final corrosion rate. Sample calculations are listed in the Appendix.

2. Results

The results of the corrosion of niobium in various concentrations of HF are summarized in Figure 14 which shows that the corrosion rate in 24 hours increased from 0 to .017, .030, .055, .090 mg./cm.^{2}hr. in 1, 2, 5, and 10 normal HF respectively. After that period the rate started to decrease and become more or less erratic during the remainder of the experiment. However, it can clearly be seen that the rate of corrosion increases with the concentration of the acid. Figure 15 shows the linear relationship between the average maximum corrosion rate and the normality of HF. Both plots reveal that niobium is very resistant to corrosion by HF (Figure 18) e.g. as compared with Fe in HF, HCl, and H_2SO_4 (Figures 19, 20, and 21). All the data are listed in the Appendix.

Still smaller are the corrosion rates of Nb in HCl and H_2SO_4. Only qualitative studies were made in these acids. These samples showed a very slight dulling of the metallic luster as evidence of the slight corrosion after two weeks.

The results of corrosion studies with hydrogen containing niobium are shown in Figures 16 and 17.

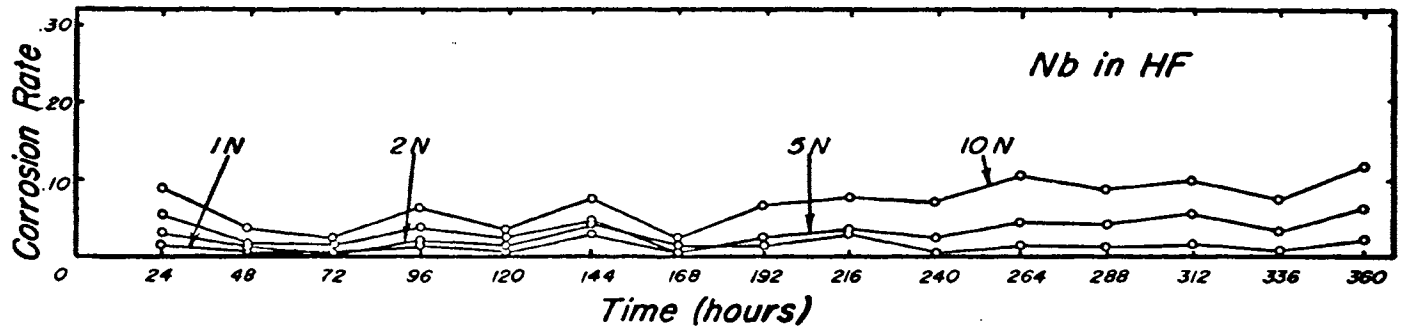

FIGURE 14. CORROSION RATE VERSUS TIME FOR Nb in HF.

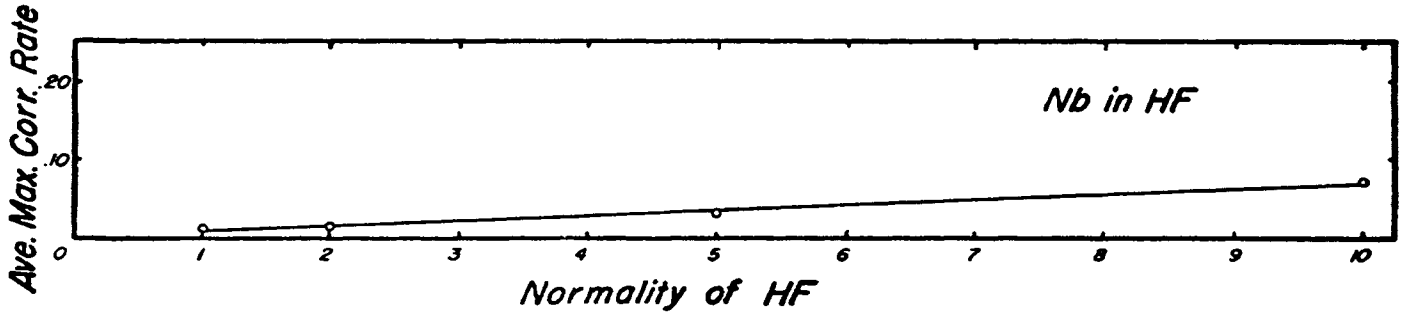

FIGURE 15. AVERAGE MAXIMUM CORROSION RATE VERSUS NORMALITY FOR Nb IN HF.

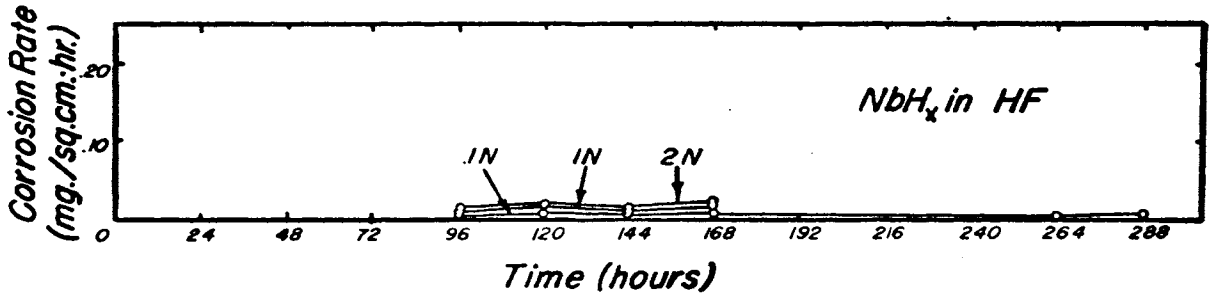

FIGURE 16. CORROSION RATE VERSUS TIME FOR NbH_x IN HF.

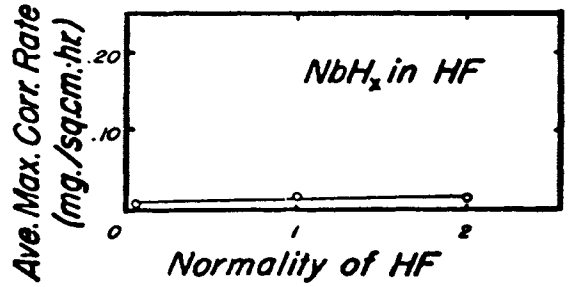

FIGURE 17. AVERAGE MAXIMUM CORROSION RATE VERSUS NORMALITY FOR NbH_x IN HF.

Reliable results were difficult to obtain with this metal in a very dilute HF and impossible to obtain in HF stronger than 2N, due to crumbling of the samples in the stronger acid during a period of less than a day. The samples in .1N, 1N, and 2N crumbled after 12, 7, and 7 days respectively. The observations were also aggravated by the very brittle nature of the samples causing them to break if not handled with utmost care. The results obtained indicate that the hydrogen content of the Nb has no great effect on the corrosion rate of the metal at least not in an acid as strong as 2N HF, except that the pieces disintegrated mechanically. In HCl and H_2SO_4 the corrosion of hydrogen containing niobium was negligible because qualitative studies in these acids showed only a darkening of the surface while two weeks under the acids.

The results of the corrosion studies on iron in HCl are shown in Figure 18, revealing that the corrosion rate was very erratic. The plot of the average maximum corrosion rate versus acid normality in HCl (Figure 21) shows however, a fair agreement with the results of Welch.[18]

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[18] *Gary E. Welch, "Dissolution Rates of Zone Refined Iron and Steel in Hydrochloric and Sulfuric Acids and in Sodium Chloride Solutions," (partially published Master's Thesis, The University of Missouri - Rolla, Rolla, Missouri, 1963), p. 48.*

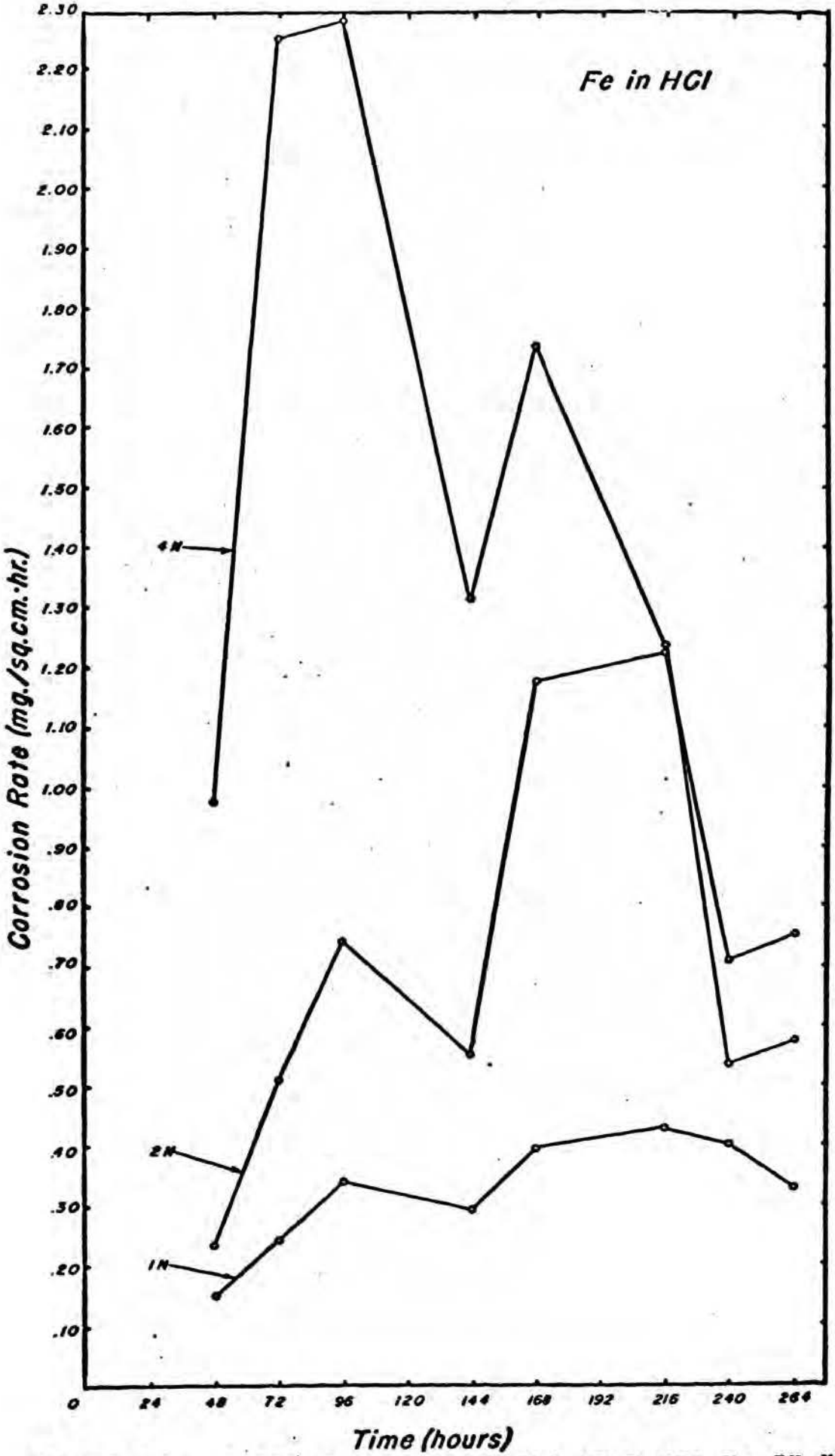

FIGURE 18. CORROSION RATE VERSUS TIME FOR Fe IN HCl.

Figure 19 represents the results of the corrosion studies on iron in H_2SO_4. The erratic nature of the corrosion rate is noted here again but, nevertheless, the plot of average maximum corrosion rate versus acid normality for H_2SO_4 in Figure 21 is also in good agreement with the findings of Welch.[19]

The results of the corrosion studies on iron in HF are summarized in Figure 20. The erratic nature of the corrosion rate here and, as given by Figures 18 and 19, is examined in the Discussion. The average maximum corrosion rate versus acid normality for HF is shown in Figure 21 but no literature was found dealing with the corrosion of iron in HF.

Figure 22 shows the results of corrosion studies on iron-niobium alloys in HF. It is immediately apparent that the addition of the Nb greatly reduces the corrosion rate of iron in HF. This plot is also much less erratic than that dealing with pure iron in HF (see Figure 20).

The results of the corrosion studies on iron-niobium alloys in 1N H_2SO_4 are shown in Figure 23. Again a marked decrease is noted in the corrosion rate and in its erratic behavior as compared to pure iron in H_2SO_4 (see Figure 19).

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[19] *Ibid.*, p. 54.

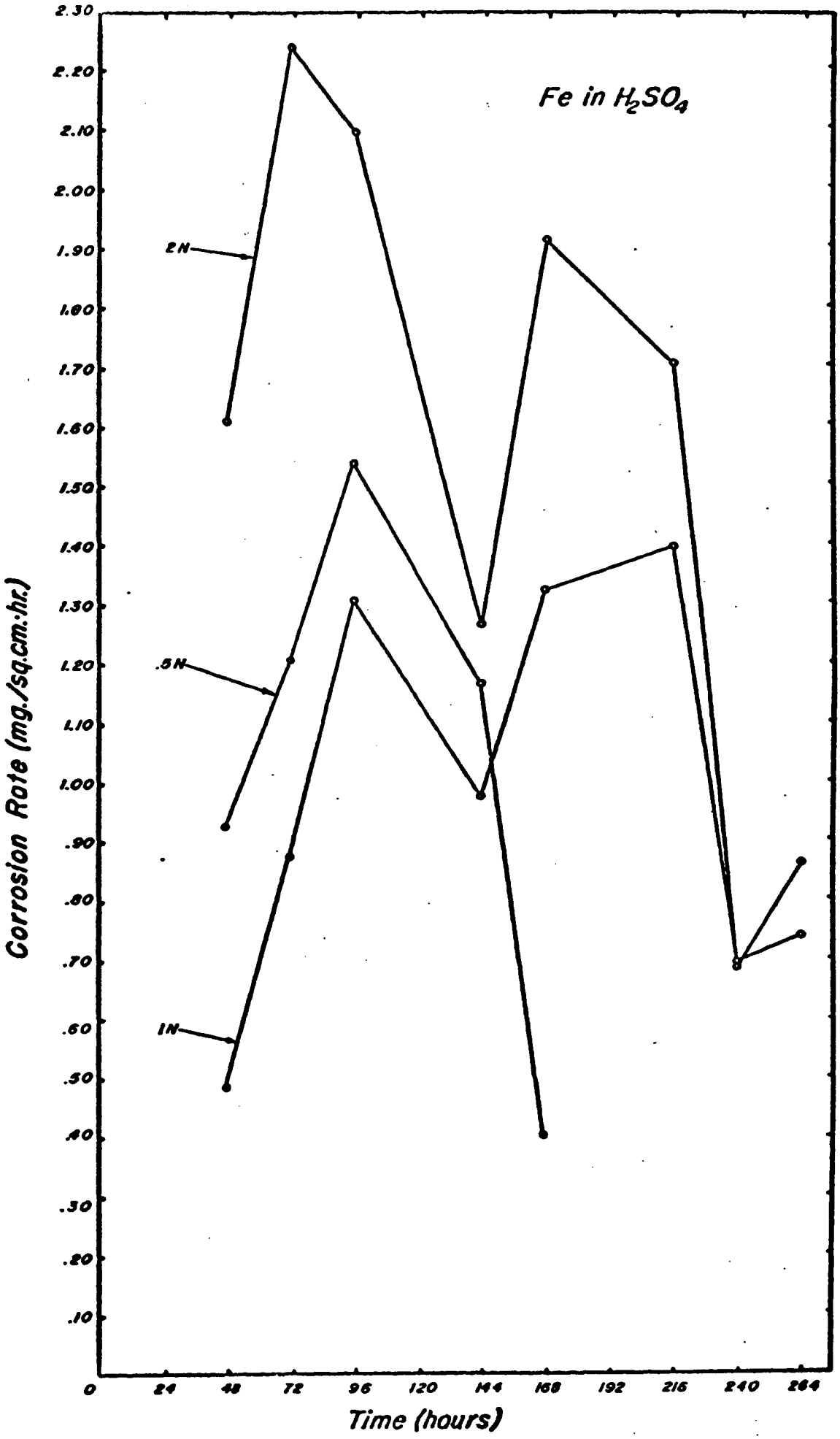

FIGURE 19. CORROSION RATE VERSUS TIME FOR Fe IN H_2SO_4.

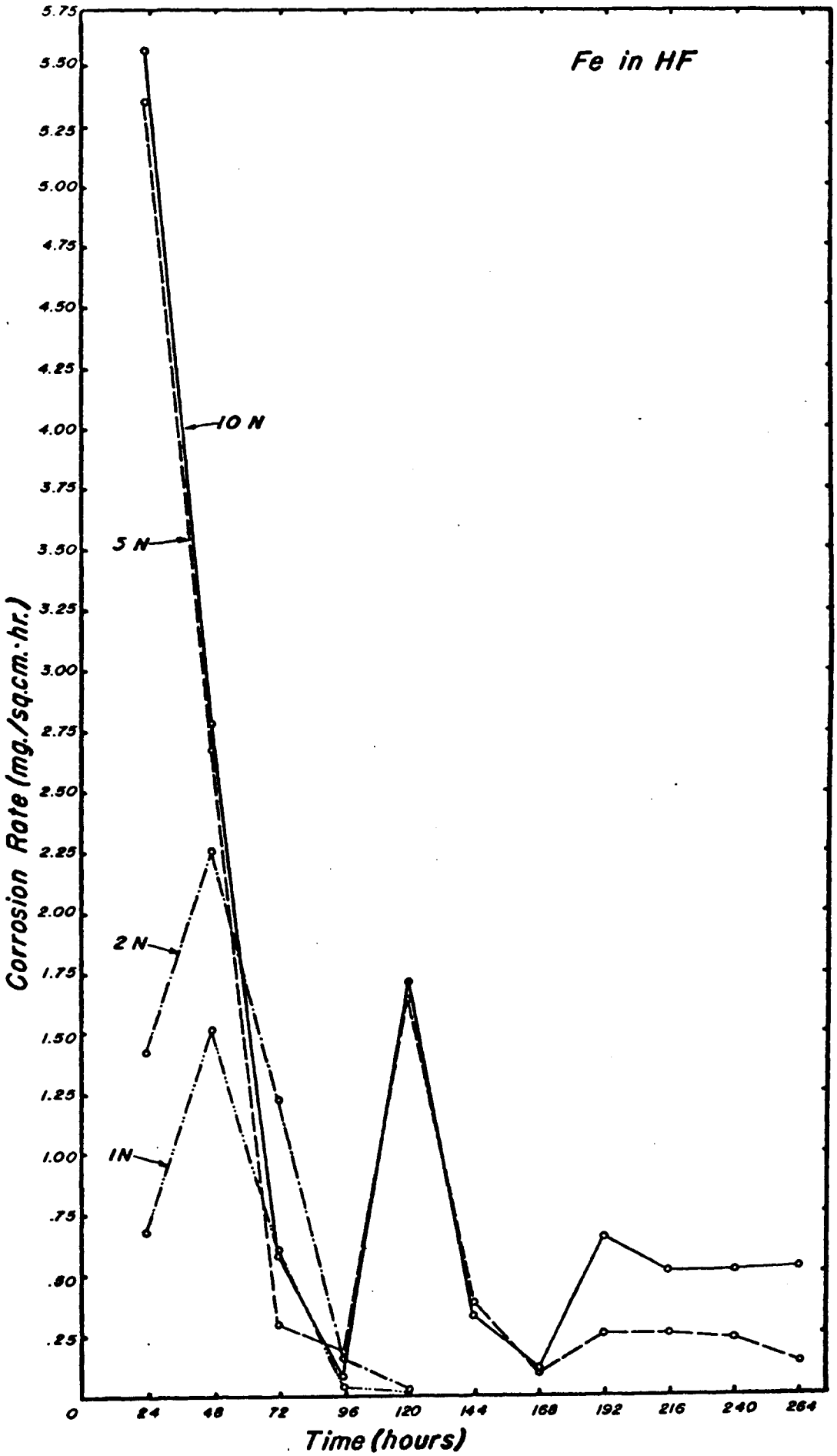

FIGURE 20. CORROSION RATE VERSUS TIME FOR Fe IN HF.

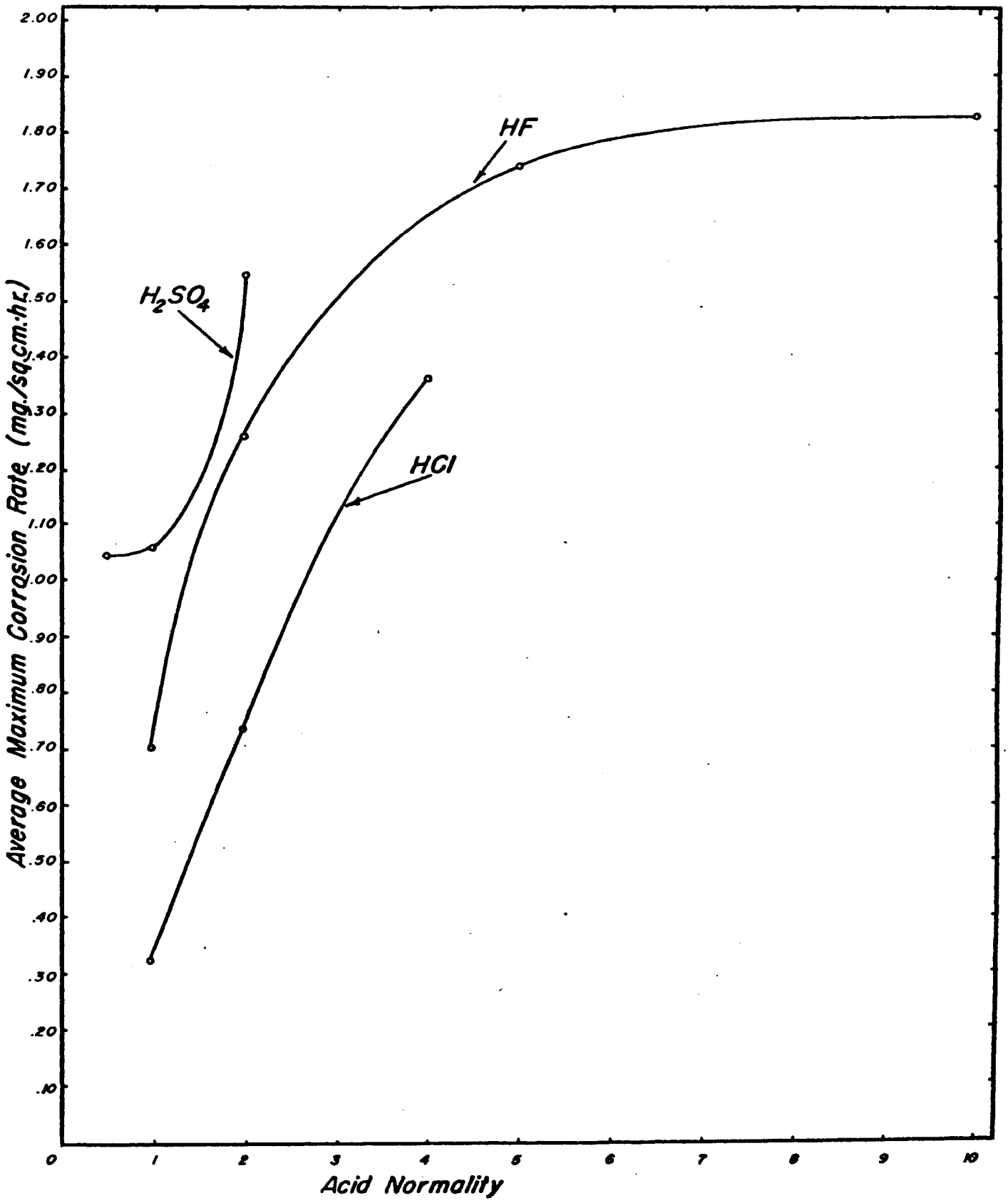

FIGURE 21. AVERAGE MAXIMUM CORROSION RATE VERSUS NORMALITY FOR Fe IN HCl, H_2SO_4, AND HF.

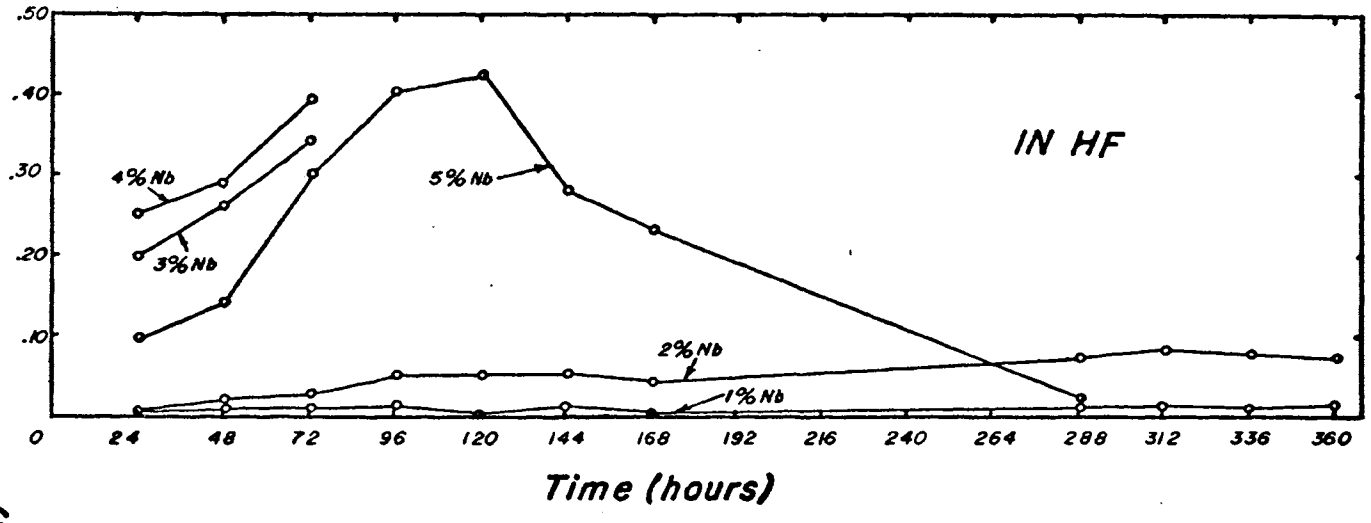

FIGURE 22. CORROSION RATE VERSUS TIME FOR Nb-Fe ALLOYS IN HF.

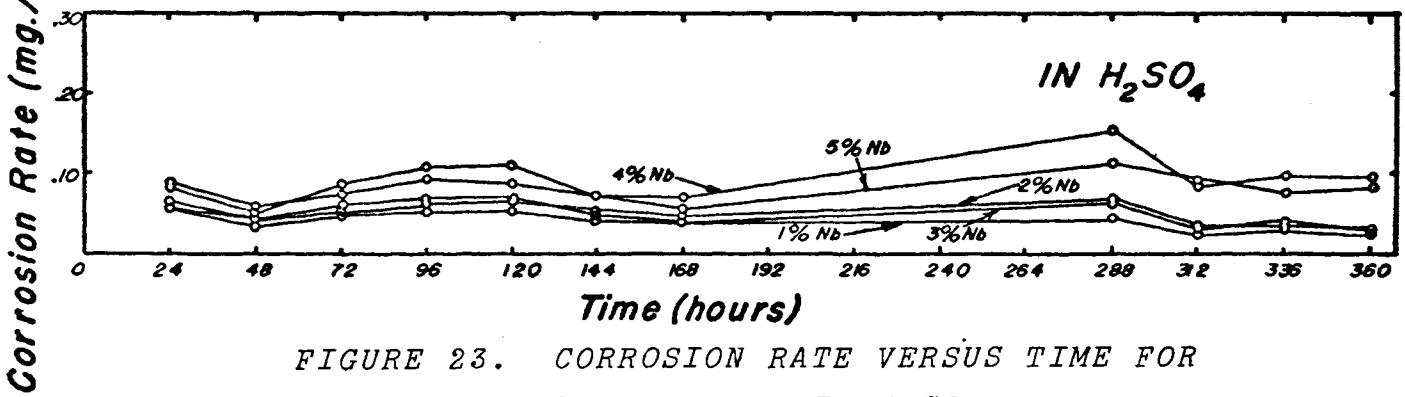

FIGURE 23. CORROSION RATE VERSUS TIME FOR Nb-Fe ALLOYS IN H_2SO_4.

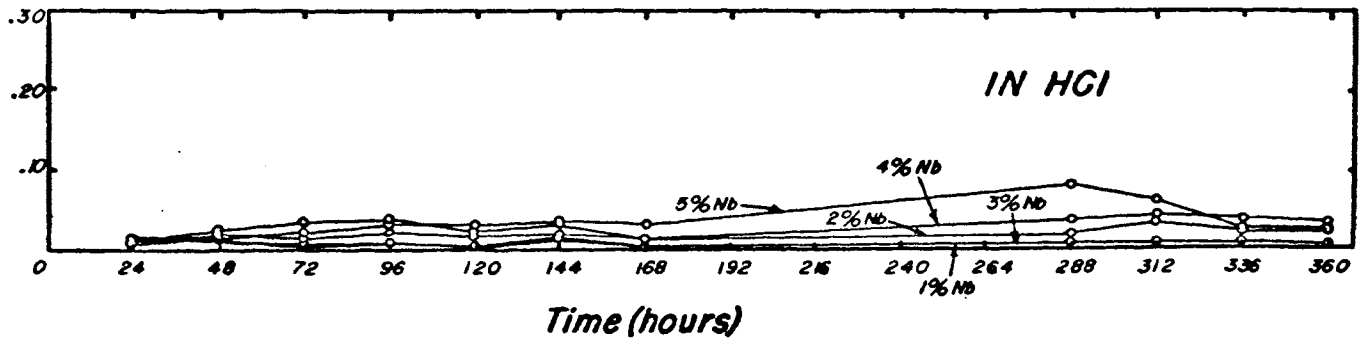

FIGURE 24. CORROSION RATE VERSUS TIME FOR Nb-Fe ALLOYS IN HCl.

The same can be stated regarding the rates obtained in 1N HCl (Figure 24, Fe in HCl see Figure 18). The plots resemble those of Figures 22 and 23. A 1% Nb addition seems to produce the largest decrease in the corrosion rate. For this reason it was decided to study the corrosion of a .5% Nb alloy.

The results of the studies of a .5% Nb - 99.5% Fe alloy, attacked by 1 and 2N HCl, H_2SO_4, and HF, are shown in Figure 25. These plots show an increase in both corrosion rate and erratic behavior over those in Figures 22, 23, and 24 meaning that addition of .5% by weight Nb is less effective in reducing the corrosion rate of iron than 1% by weight Nb.

Figure 26 shows a plot of the average maximum corrosion rate versus percent niobium in 1N HF, H_2SO_4, and HCl. Clearly, the corrosion rate reaches a minimum between 1 - 1.5% by weight Nb added to the Fe.

D. INVESTIGATION OF SKIN FORMATION

When the studies were begun on the corrosion rates of iron and iron-niobium alloys the first samples were made using the iron powder just as it came from the supplier. They were hung in the acids and weighed each day as described above. However, after three or four days they began to gain weight and upon examination an acid resistive skin was found which covered all surfaces of the samples. The skin could be peeled off with a

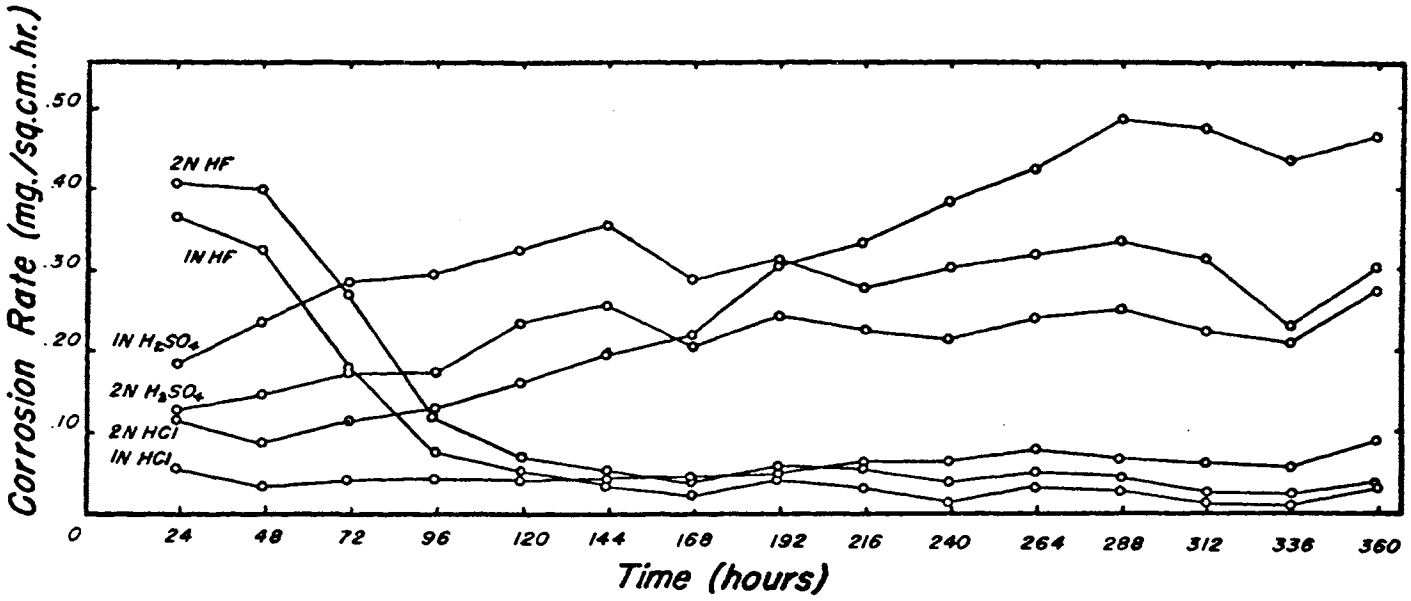

FIGURE 25. CORROSION RATE VERSUS TIME FOR .5%Nb-99.5%Fe IN HCl, H_2SO_4 AND HF.

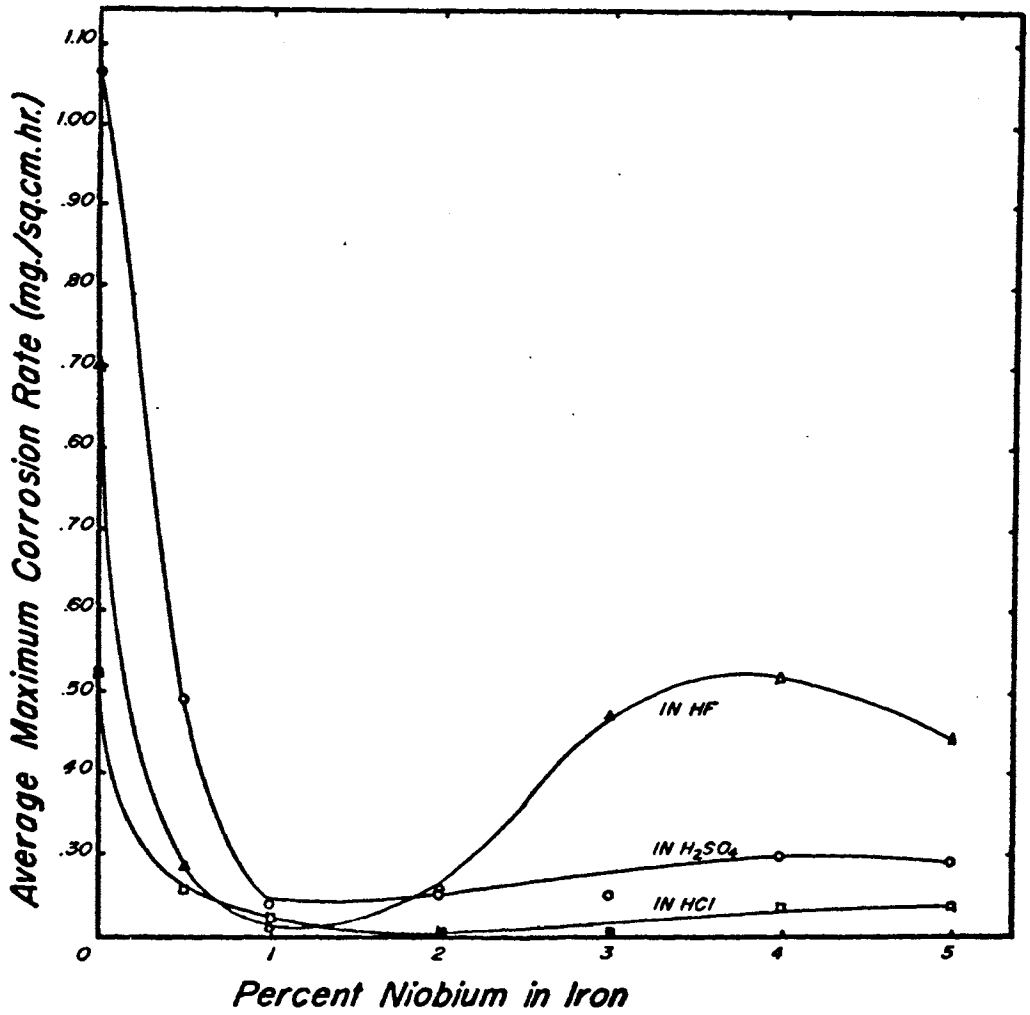

FIGURE 26. AVERAGE MAXIMUM CORROSION RATE VERSUS PERCENT NIOBIUM FOR Fe-Nb ALLOYS IN 1N HCl, H_2SO_4, AND HF.

sharp blade and a very rough and corroded core appeared. Figure 27 shows the external surface of a pure iron sample which was corroded for two weeks in 2N HCl. The skin is still intact in this figure. The top skin is partially removed in Figure 28 and completely removed in Figure 29. The core with the skin completely removed and the inside of the top skin are shown in Figures 30 and 31 respectively. Photomicrographs at different magnifications of a cross section through a sample with the skin intact are shown in Figures 32 and 33. In these figures pores penetrating the skin are easily recognizable. These pores are also shown in Figures 34 and 35 where the former represents a portion of the skin by reflected light and the latter by transmitted light. Figures 36 and 37 show the same as Figures 34 and 35 except that the skin pictured was formed in four days of corrosion in 2N HCl.

It was found that by heating the iron powder to 600°C for three hours in a stream of hydrogen and cooling it in the gas before pressing the samples, the skin would not form. To find the reason for this behavior, samples of powder "as received" and "H_2 treated" were subjected to carbon analysis by the chromatographic method. It revealed that the "as received" powder contained 0.013% carbon while the "H_2 treated powder

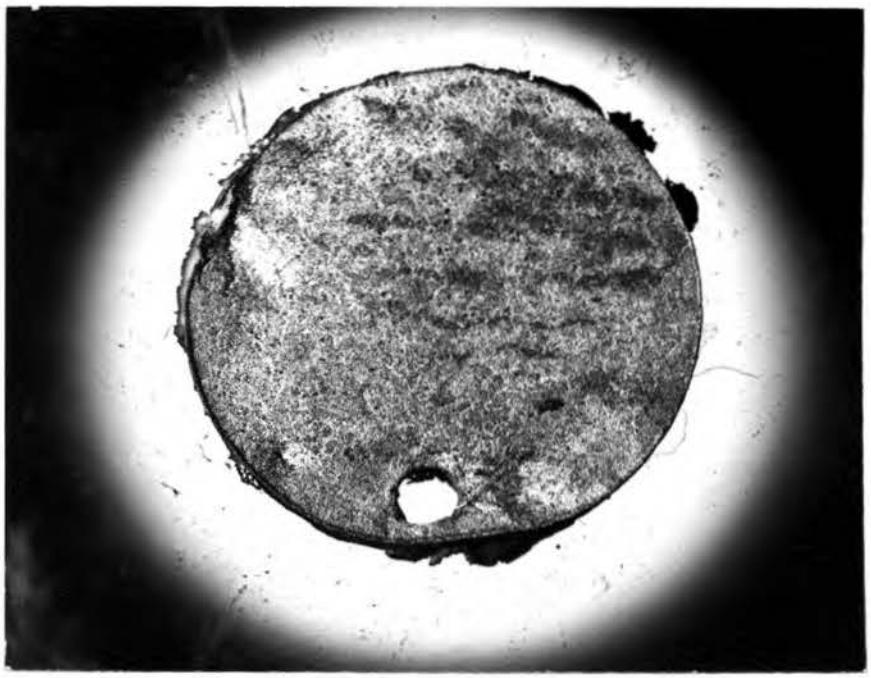

FIGURE 27

Pure iron sample, no H_2 treatment, corroded two weeks in 2N HCl forming a skin. This is the outside of the sample before the skin is removed. (5X)

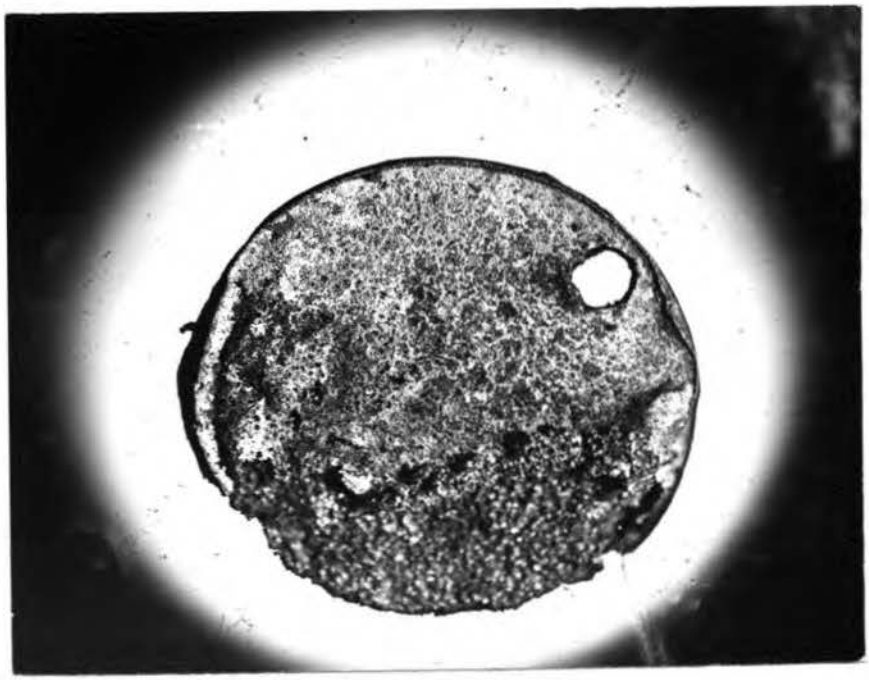

FIGURE 28

Pure iron sample, no H_2 treatment, corroded two weeks in 2N HCl forming a skin. This shows the skin partially removed from the core which is out of focus. (5X)

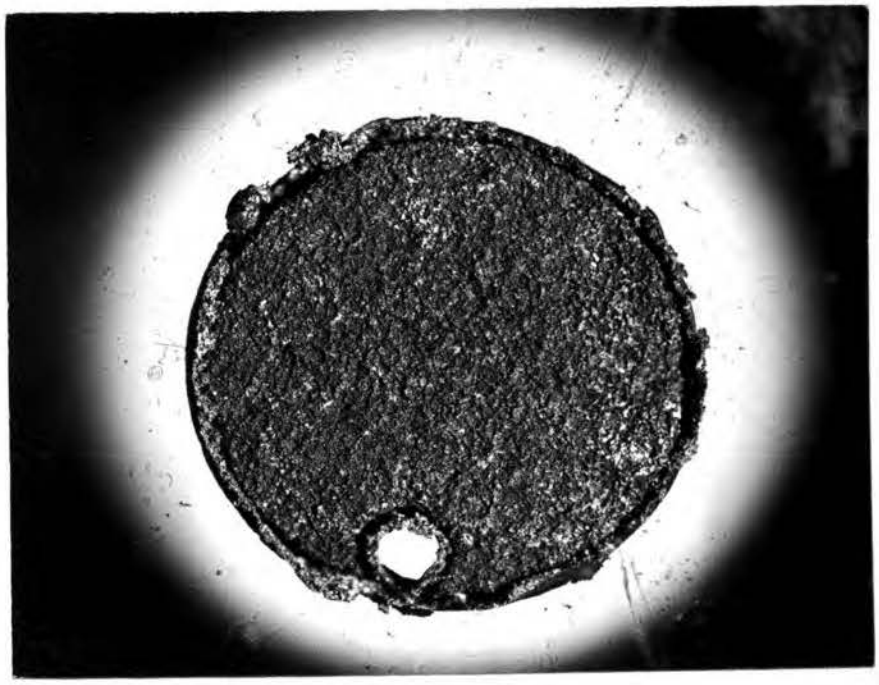

FIGURE 29

Pure iron sample, no H_2 treatment, corroded two weeks in 2N HCl forming a skin, which is removed. The lateral skin is still present. (5X)

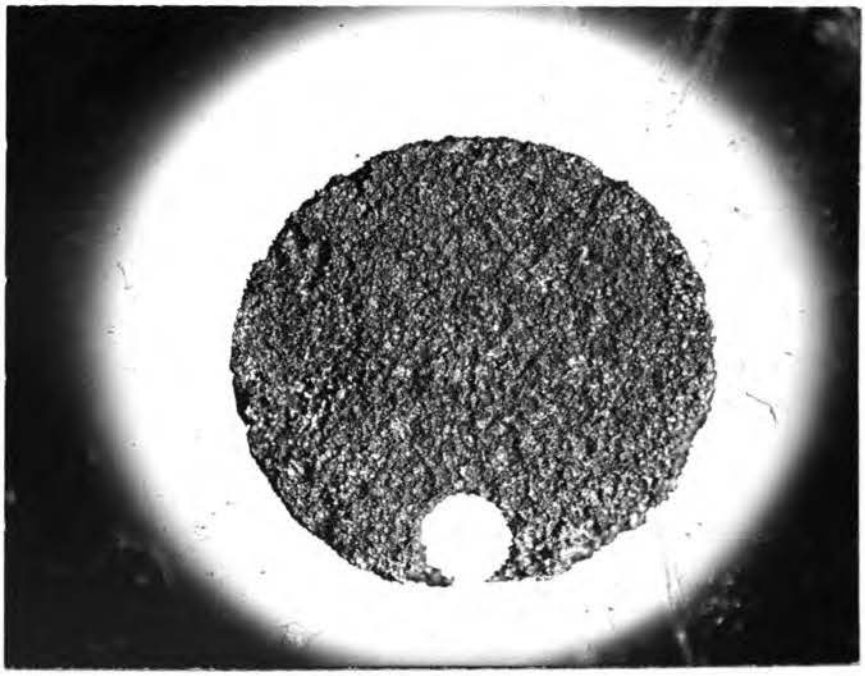

FIGURE 30

Pure iron sample, no H_2 treatment, corroded two weeks in 2N HCl forming a skin, which is completely removed, only the core remained. (5X)

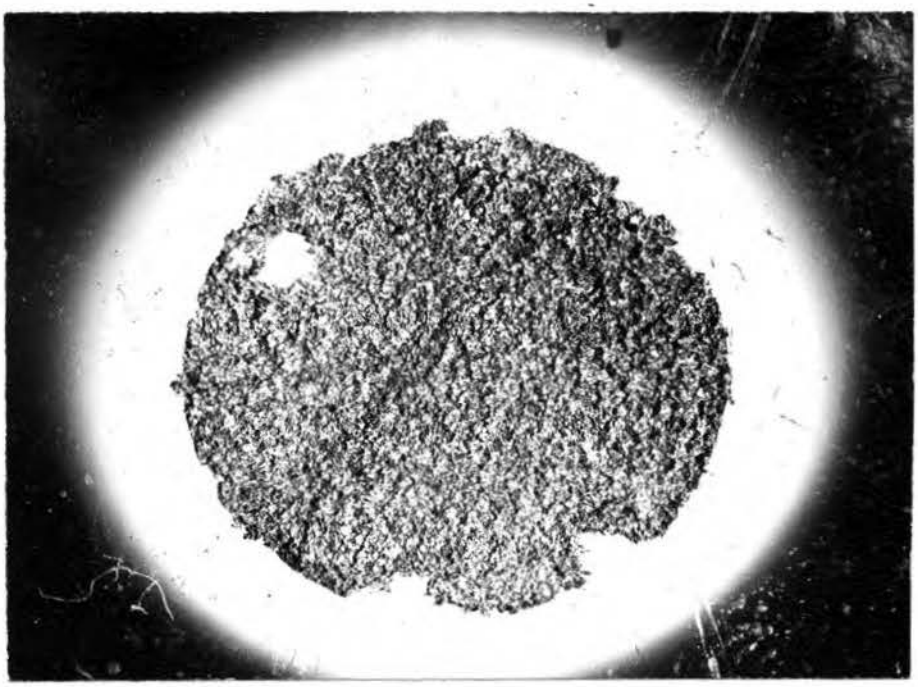

FIGURE 31

Pure iron sample, no H_2 treatment, corroded two weeks in 2N HCl forming a skin. This is the underside of the skin after it was peeled off the core. (5X)

FIGURE 32

Pure iron samples, no H_2 treatment, corroded two weeks in 2N HCl forming a skin. This is a cross section of skin and core intact. (25X-nital etch)

FIGURE 33

Pure iron sample, no H_2 treatment, corroded two weeks in 2N HCl forming a skin, This is a cross section of skin and core intact. (75X-nital etch)

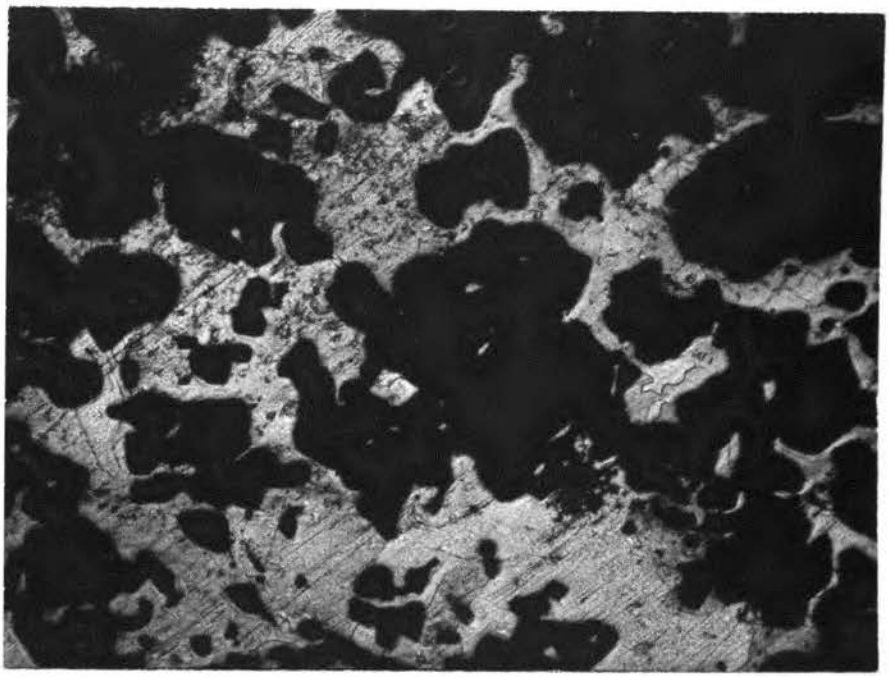

FIGURE 34

Pure iron sample, no H_2 treatment, corroded two weeks in 2N HCl forming a skin. This shows a portion of the skin in reflected light. (75X)

FIGURE 35

Pure iron sample, no H_2 treatment, corroded two weeks in 2N HCl forming a skin. This shows the same field as Figure 34 in transmitted light. (75X) Pores-bright

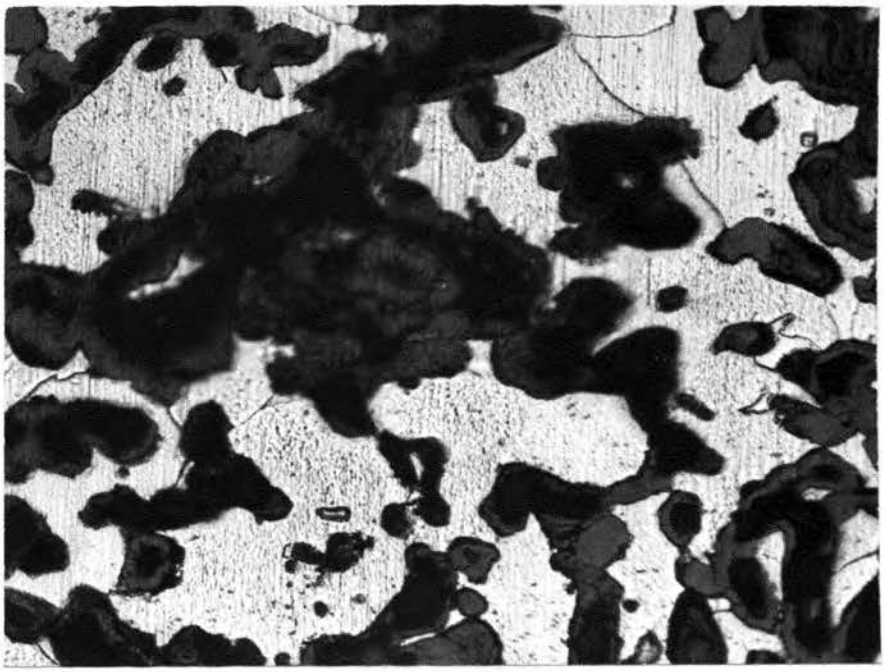

FIGURE 36

Pure iron sample, no H_2 treatment, corroded four days in 2N HCl forming a skin. Portion of the skin in reflected light. (500X-nital etch)

FIGURE 37

Pure iron sample, no H_2 treatment, corroded four days in 2N HCl forming a skin. Pore in it, transmitted light, (500X)

had only 0.0035%. It is reported by Chappell,[20] and Aitchison[21] that the carbon content has a definite effect on the corrosion rate of iron. They both report that the corrosion rate increases with increasing carbon content.

On the basis of the carbon analysis and the work of Chappell and Aitchison it is possible to explain how the skin is formed. When the first samples were pressed from the "as received" powder and sintered at 1350°C for two hours under hydrogen, the carbon in the outer surface of the samples diffused out and was carried away by the hydrogen, probably as CH_4, leaving the outer surface to a depth of about .10 to .12mm. with much less carbon than was in the core. Therefore, when the samples were hung into the acid the pores in the sample allowed the acid to penetrate below the carbon poor surface layer and corrode the core, which due to its higher carbon content, corroded faster than the surface layer. The acid eventually corroded away the iron between the surface layer and the core, thereby producing the loose surface Fe layer -- the skin on the samples. The

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[20] C. Chappell, "The Influence of Carbon on the Corrodibility of Iron," Journal of the Iron and Steel Institute, LXXXV (1912), pp. 270 - 296.

[21] L. Aitchison, "Experiments on the Influence of Composition Upon the Corrosion of Steel," Transactions of the Faraday Society, XI (1915), pp. 212 - 234.

hydrogen liberated in the reaction

$$Fe + 2HCl = FeCl_2 + H_2$$

inflated the corroding sample. This explanation is further substantiated by the fact that the skin was thicker and it took longer to form when a sample made from "as received" powder was inadvertently sintered for more than two hours. Thus, the carbon was able to diffuse at 1350°C from a greater depth due to the longer time. Theoretically, it would seem that if the samples were sintered long enough there would be no skin effect. This was essentially achieved by treating the powder at 600°C under hydrogen before pressing the sample. All the carbon that was able to diffuse out, did so during the pretreatment. When finally the samples were sintered there was little carbon left. Even if this amount would leave the surface layer, no significant difference in the carbon content of the surface and the core would result. Therefore, the acid did not attack the core preferentially through the pores.

E. ELECTROCHEMICAL POTENTIALS OF Fe, Fe - Nb ALLOYS, Nb, AND NbH_x IN HCl, H_2SO_4, AND HF

1. Apparatus and Procedure

To determine the potentials of the various samples they were first mounted in lucite of about one centimeter thickness. A hole was then drilled through the back of

the lucite until the drill produced a small cavity in the back of the metal. A small glass tube 4 mm. in diameter and about 15 cm. long with two centimeters bent at 90° on one end was inserted with this end into the hole in the lucite as shown in Figure 38. The gap between the lucite and the tubing was sealed with epoxy cement and a drop of mercury was put down the glass tube so that it would touch the back of the metal. This was to form a good contact between the metal and a copper wire which was run down the glass tube into the mercury. The other electrode was a normal calomel standard half-cell. A 3.5 N KCl salt bridge connecting the half-cell with the electrode eliminated the junction potential. The complete arrangement of half-cells and salt bridge is shown in Figure 38. Plastic tubing and a plastic bottle were substituted for the glass for work with HF.

To measure the potential of an electrode, the metallic end of it was first ground smooth on 600 grit emery paper and then the glass shaft of the electrode was inserted through the stopper in the top of the electrolyte container. The electrode was first located above the level of the electrolyte until the container was purged with argon. Then the electrode was pushed down into the electrolyte and adjusted so that the end of the capillary tube was against the metal sample as

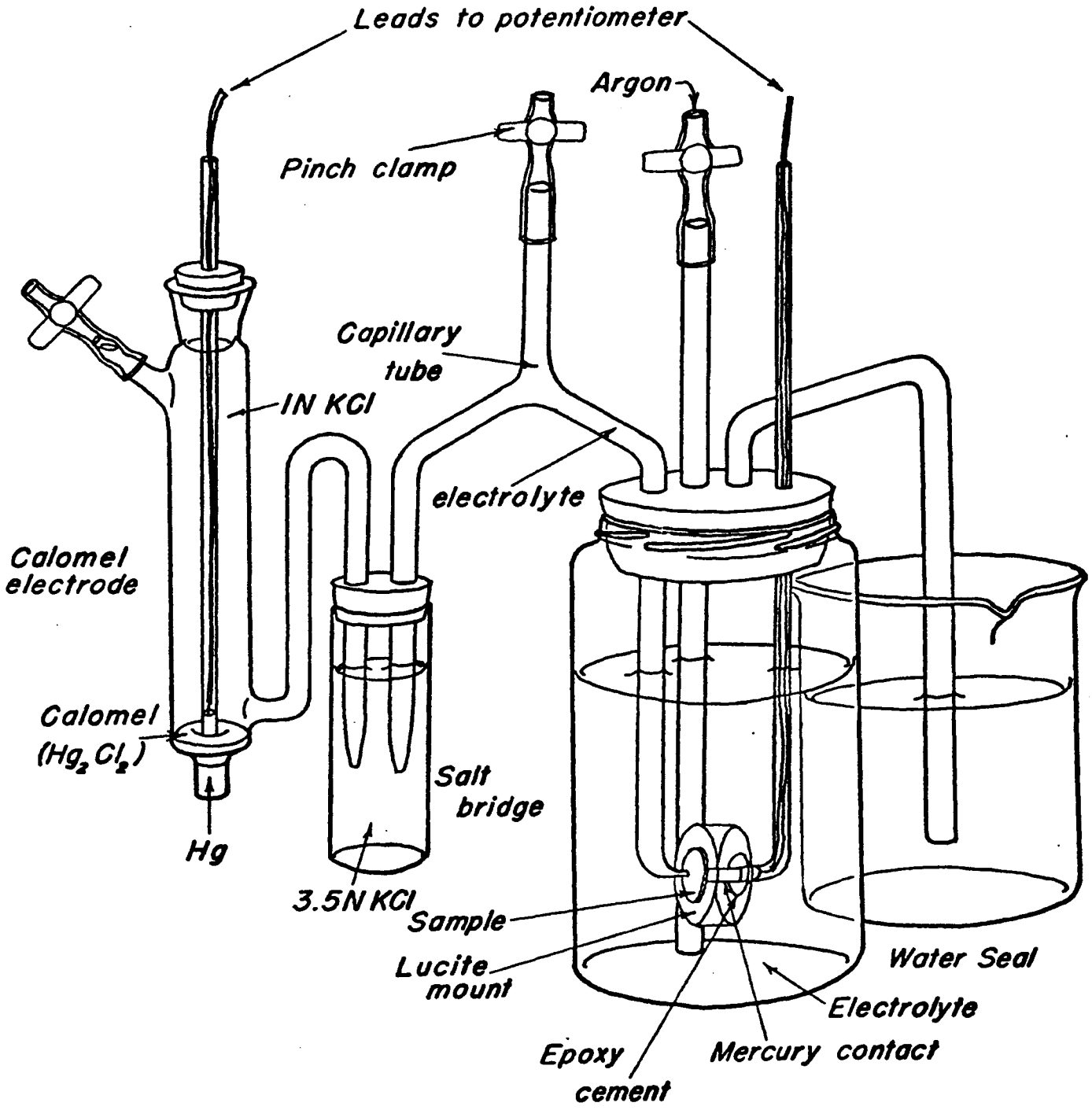

FIGURE 38. APPARATUS USED FOR THE DETERMINATION OF THE ELECTROCHEMICAL POTENTIALS OF Fe, Fe-Nb ALLOYS, Nb, AND NbH_x IN HCl, H_2SO_4 AND HF.

shown in Figure 38. Potential measurements were taken, as soon as the electrode was in place, and every minute for the first ten minutes. Then readings were taken every five minutes for twenty minutes and finally every ten minutes until the readings became constant. Argon was bubbled through the electrolyte throughout the time of the measurements. The measurements in HF were taken every minute until a constant reading was reached.

The potentiometer readings were converted to the hydrogen scale by algebraic addition of the potential of the calomel half-cell against the 1N hydrogen electrode. This was approximately +0.281 volts, the reference electrode being standardized against a saturated calomel electrode before use. Sample calculations and all data appear in the Appendix. The potentiometer was frequently checked with a zinc electrode to be sure of proper polarity and to check the instruments batteries.

2. Results

Figure 39 is a plot of potential versus time for the 99% Fe - 1% Nb alloy dissolving in HCl and H_2SO_4. It is typical of all the potential versus time curves, the remainder of which are in the Appendix. Some of the plots are fairly erratic but they all eventually level off. Another common point is that the rate of change of the potential is greatest at the very beginning of

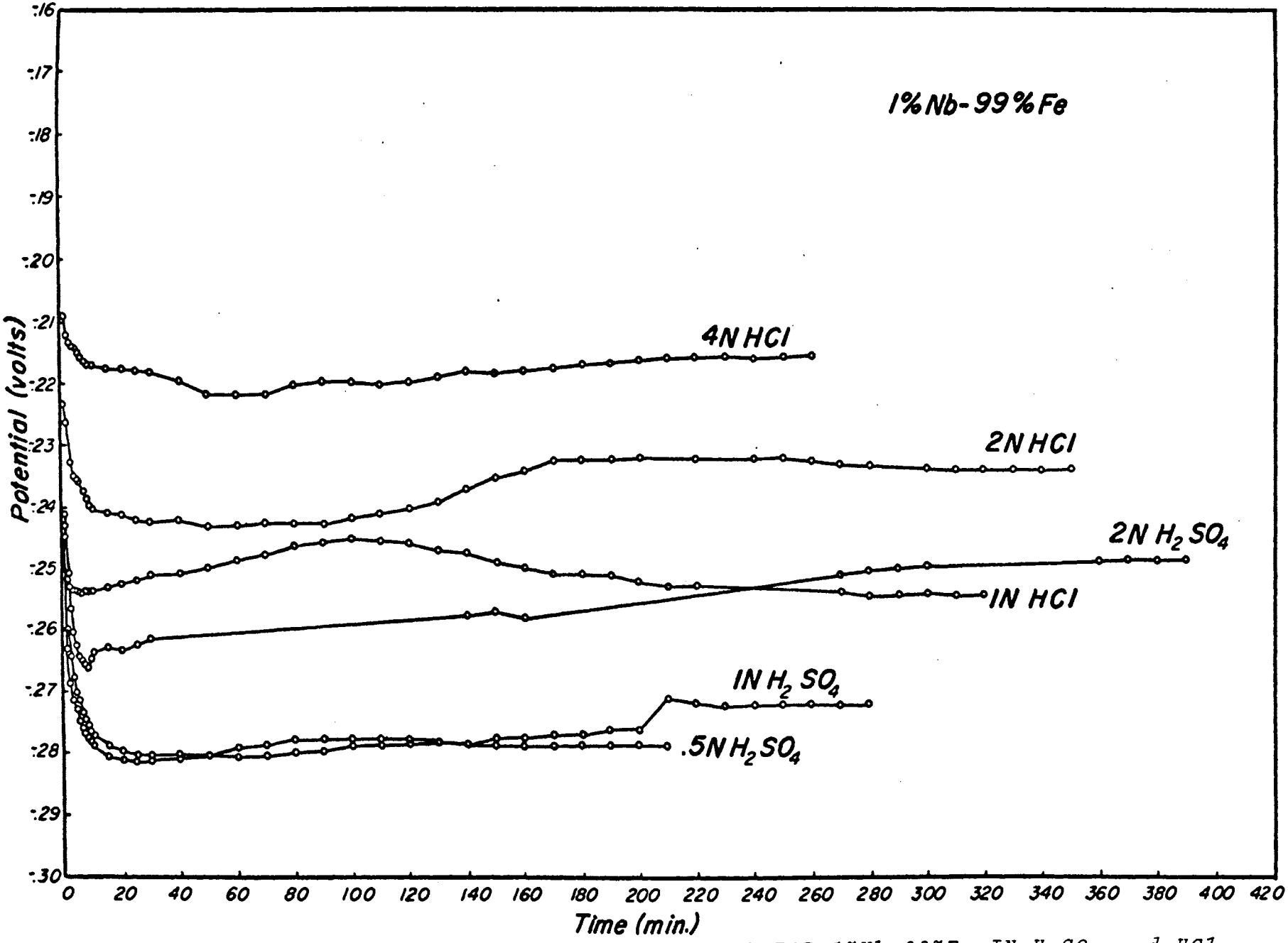

FIGURE 39. THE PLOTS OF POTENTIAL VERSUS TIME FOR 1%Nb-99%Fe IN H_2SO_4 and HCl.

each run, usually within the first five minutes.

The electrochemical potentials of Fe and Fe - Nb alloy electrodes immersed in .5, 1, and 2N H_2SO_4 are summarized in Figure 40 which shows the relationship between the steady potential reached by each sample and the weight percent of Nb in each sample. Figure 41 is a similar plot for Fe and Fe - Nb alloys in 1, 2, and 4N HCl. Figure 42 shows the steady potentials in .1, 1, 2, and 5N HF. These plots are similar in that they show a generally more positive potential with an increasing weight percent Nb. Reproducibility was good in a general nature. That is, the general shape of the curves were the same and the steady potentials were within .02 volts of each other consistently. Generally, only two runs were made for each alloy in each acid. More runs were made when discrepencies arose or when there was a malfunction in the experiment.

Figure 43 is a plot of the steady potential versus the normality of H_2SO_4 for Fe and Fe - Nb alloys. Figure 44 is the steady potential versus the normality of HCl for these same metals. Normality of HF versus steady potential for Fe and Fe - Nb alloys is plotted in Figure 45. These three plots are similar in that they all show in general an increasingly positive steady potential for each sample with increasing normality

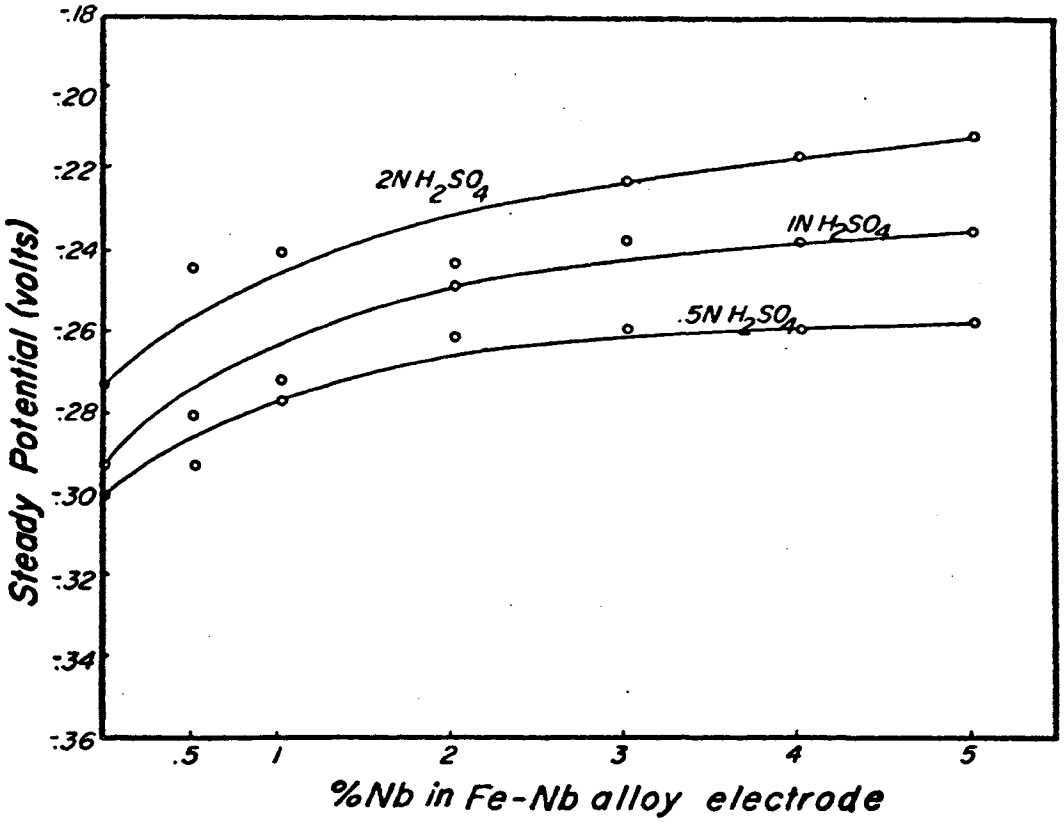

FIGURE 40. STEADY POTENTIAL VERSUS WEIGHT PERCENT Nb IN THE ELECTRODE FOR .5, 1, AND 2N H_2SO_4.

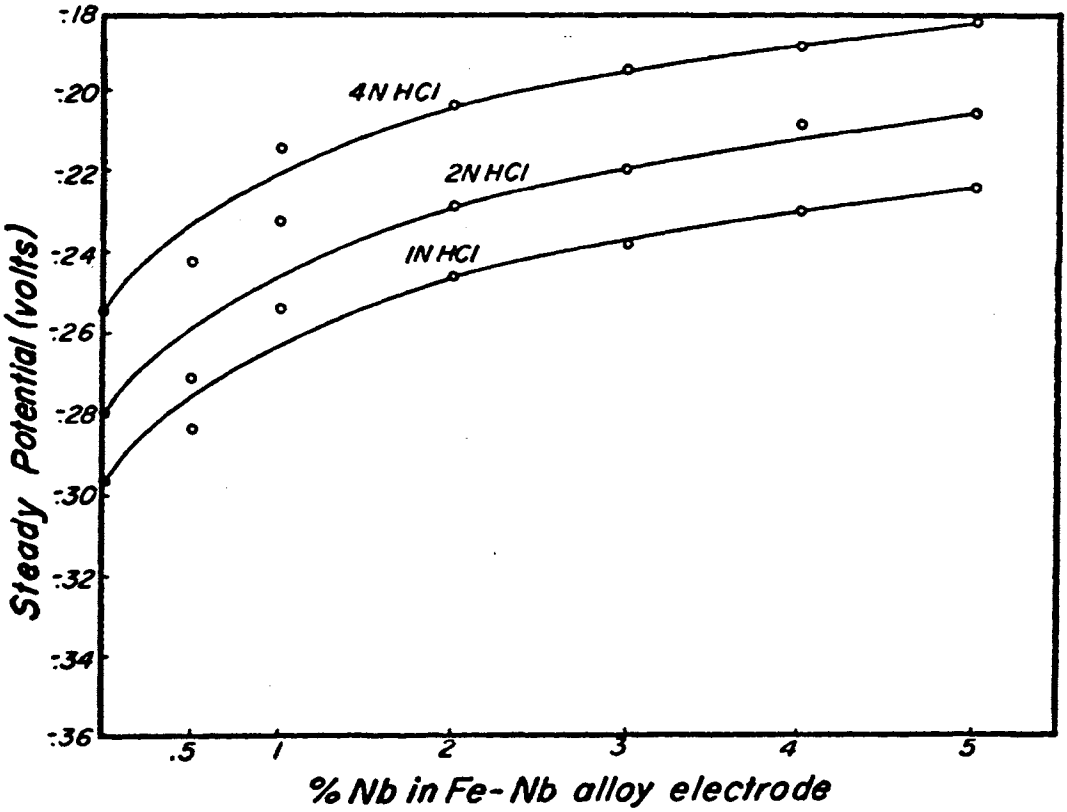

FIGURE 41. STEADY POTENTIAL VERSUS WEIGHT PERCENT Nb IN THE ELECTRODE FOR 1, 2, AND 4N HCl.

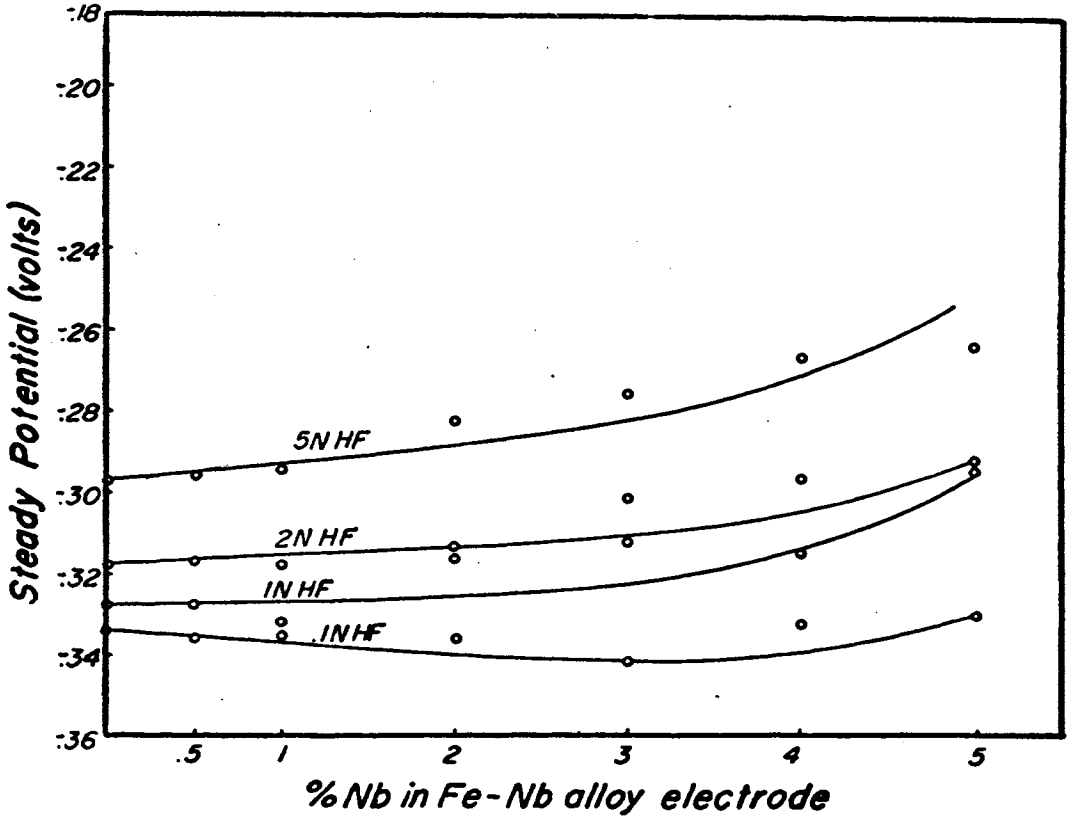

FIGURE 42. STEADY POTENTIAL VERSUS WEIGHT PERCENT Nb IN THE ELECTRODE FOR .1, 1, 2, AND 5N HF.

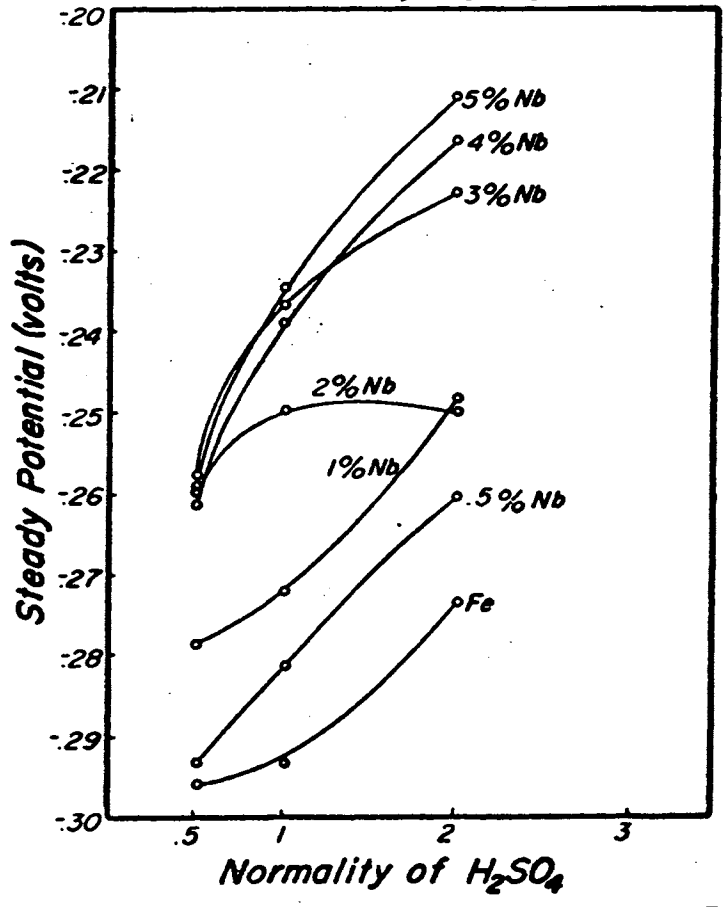

FIGURE 43. STEADY POTENTIAL VERSUS NORMALITY FOR Fe AND Fe-Nb ALLOYS IN H_2SO_4.

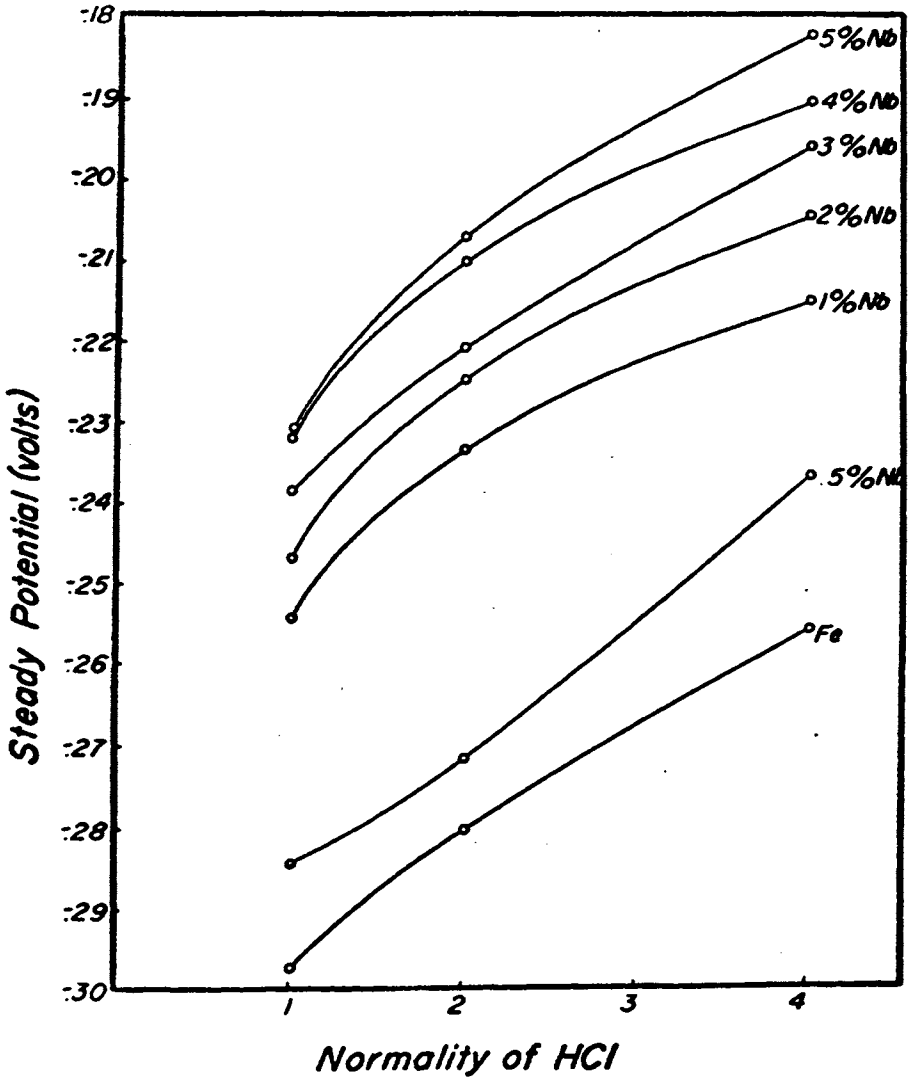

FIGURE 44. STEADY POTENTIAL VERSUS NORMALITY FOR Fe AND Fe-Nb ALLOYS IN HCl.

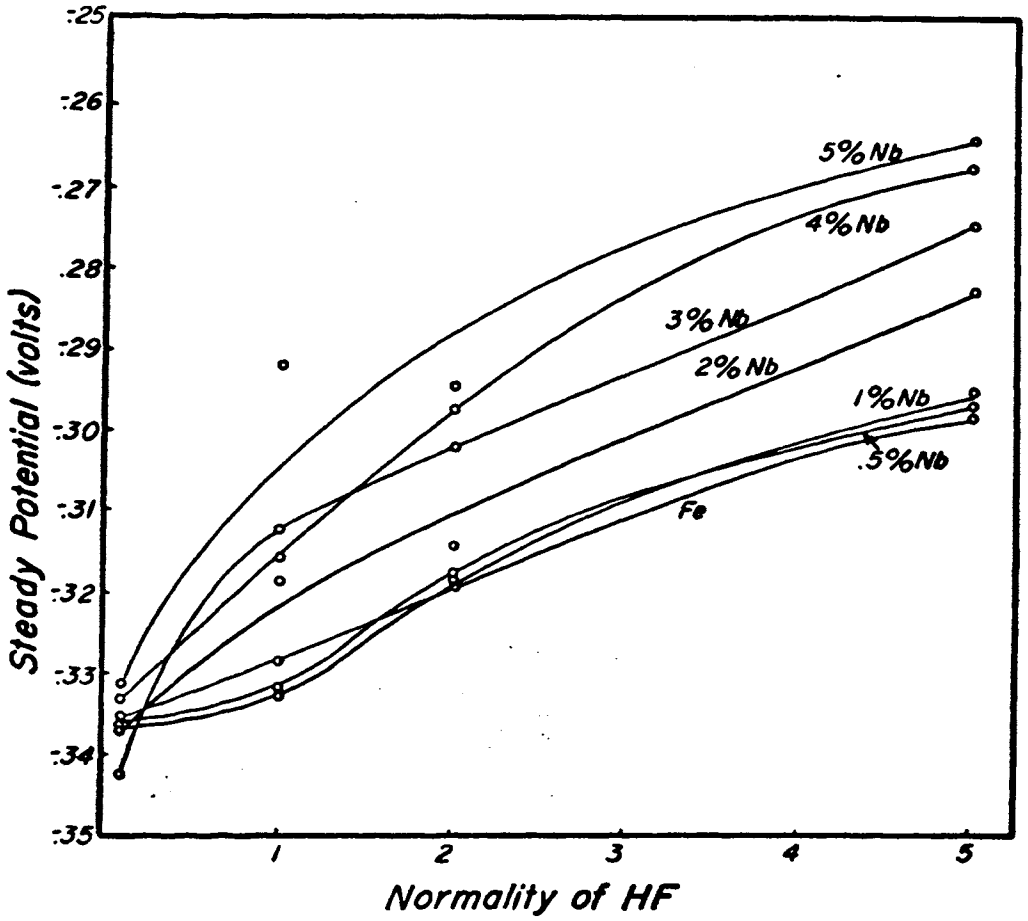

FIGURE 45. STEADY POTENTIAL VERSUS NORMALITY FOR Fe AND Fe-Nb ALLOYS IN HF.

of the acid. This agrees with behavior reported by Evans.[22]

Figure 46 is a plot of the electrochemical runs of potential versus time for both Nb and NbH_x. There is a similarity with runs of Fe - Nb alloys in HF as they also level off in less than twenty minutes. The determinations of the electrochemical potentials of Nb and NbH_x in HF are summarized in Figure 47. It shows that although there is a great change in the steady potential in low normality HF, it appears to reach a constant potential with increasing normality at about 6N. It is also shown that the potentials become more negative with increasing concentration of the HF. This is just opposite to the behavior of Fe and the Fe - Nb alloys but it is very similar to that shown by titanium[23] and other metals which have a very adherent oxide film.

The determination of the electrochemical potentials of Nb and NbH_x in H_2SO_4 and HCl were terminated because the potentials did not level off after two weeks in the acids.

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[22] Ulick R. Evans, An Introduction to Metallic Corrosion, (London: Edward Arnold Ltd., 1963), p. 235.

[23] M. E. Straumanis and P. C. Chen, "The Corrosion of Titanium in Acids," Corrosion, VII (July, 1951), pp. 229 -237.

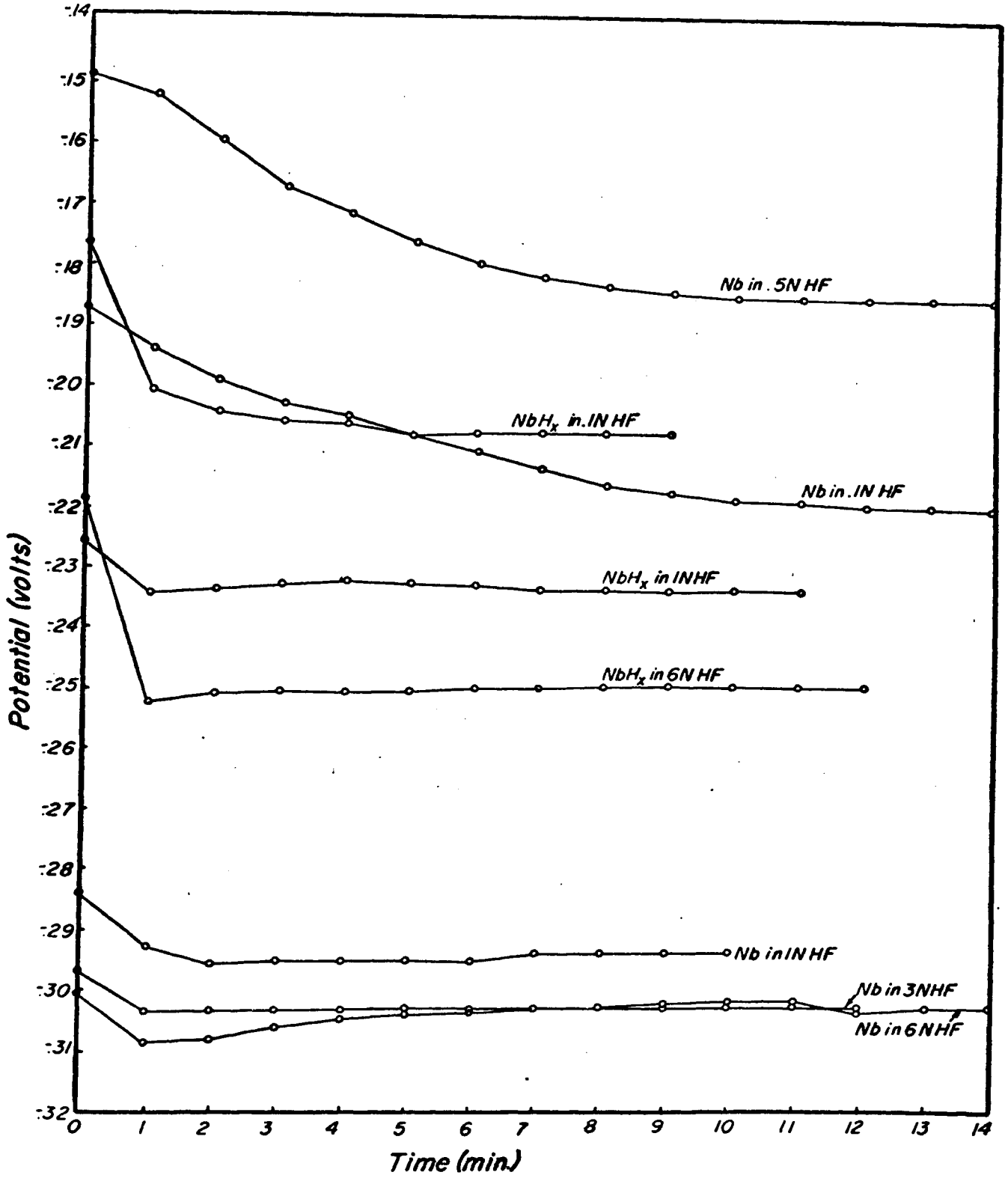

FIGURE 46. PLOTS OF POTENTIAL VERSUS TIME FOR Nb AND NbH_x IN HF.

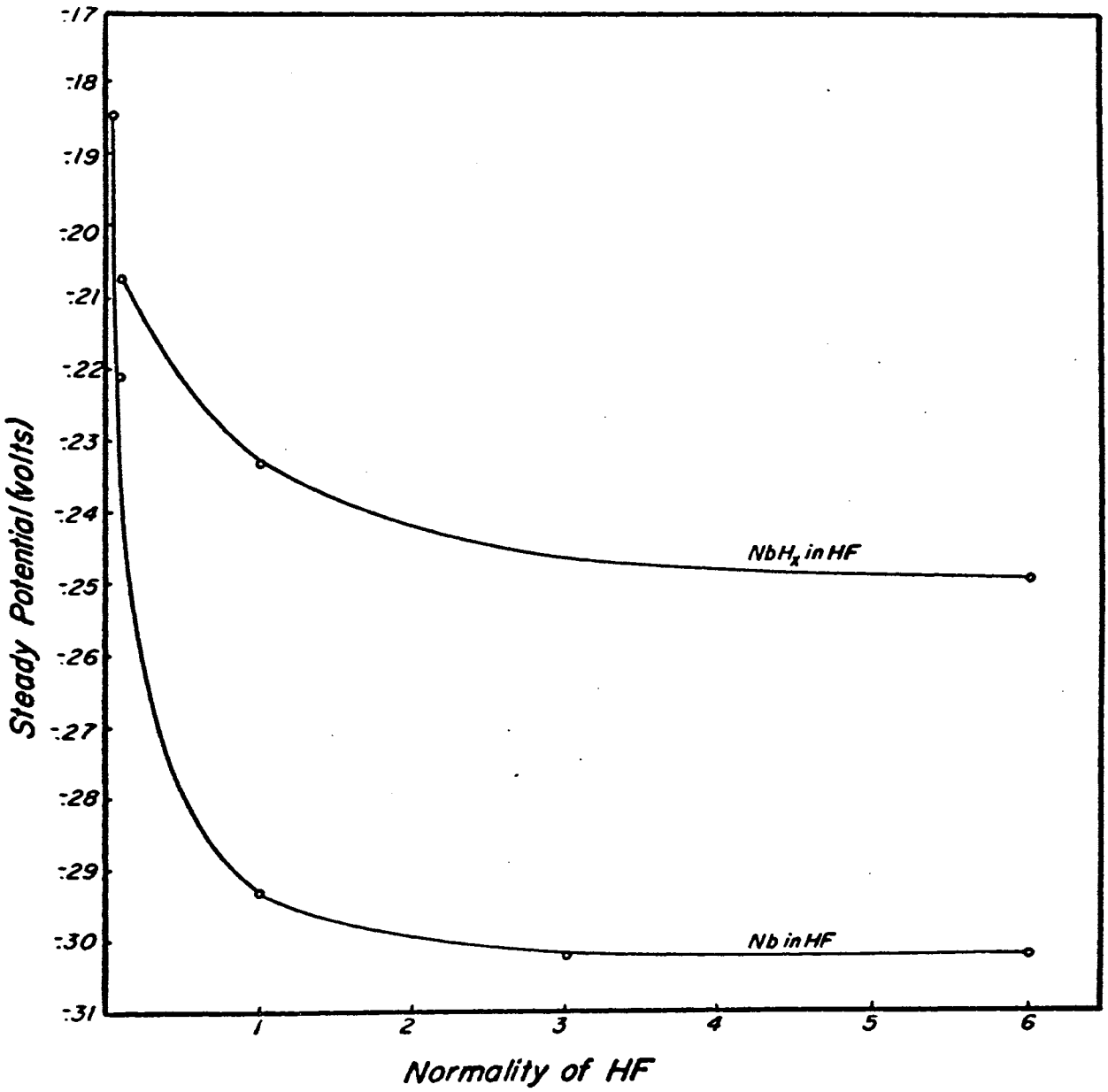

FIGURE 47. STEADY POTENTIAL VERSUS NORMALITY FOR Nb AND NbH_x IN HF.

IV. DISCUSSION

A. VALENCE OF NIOBIUM

Valences of +3, +4 and +5 have been reported for niobium and there is some speculation that a Nb^{+2} oxidation state may also exist.[24] The +5 state is the most common one, which agrees with the value obtained in this study. Nb_2O_5, niobic oxide, containing the Nb^{+5}, is the most common and stable oxide of niobium. NbO_2 is known but is most difficult to prepare and Nb_2O_3 is obtained by reducing the Nb_2O_5. No information could be found concerning the valence of Nb ions going into solution in hot HF, or in other acids. However, Remy[25] mentions NbF_5 in agreement with the valence state noted in the HF experiments.

B. CORROSION RATES IN RELATION TO ELECTROCHEMICAL POTENTIALS

It is generally true that the electrochemical potential of a metal becomes more positive with increasing rates of corrosion or decreasing pH under identical

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[24] W. L. Latimer, The Oxidation States of the Elements and Their Potentials in Aqueous Solutions (New York: Prentice-Hall, Inc., 1952), pp. 262-263.

[25] H. Remy, Treatise on Inorganic Chemistry (New York: Elsevier Publishing Co., 1956), p. 110.

conditions.[26] This was found to be true with Nb and the Nb - Fe alloys also.

This high resistance to corrosion of niobium is probably due to the formation of a coherent oxide (Nb_2O_5) film on the metal.[27] This film is formed very rapidly in air and serves to isolate the metal from the atmosphere and the acid. Nb_2O_5 is insoluble in most acids,[28] and can therefore protect the metal very well. The fact that the standard electrode potential of Nb is -1.1 [29] volts is further evidence that there must be a protective film on the metal for this potential is more active even than that of zinc, and yet zinc dissolves rapidly in any of the acids in which Nb was tried. The comparatively noble potentials which were observed in the present study, during the dissolution of Nb in HF also indicate that a protective film is present on the surface of the metal. The fact that HF attacks the Nb to a greater extent than other acids indicated that the protective film is partially dissolved by the HF and NbF_5 is formed as the Nb is dissolved in HF. There is

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[26] Evans, loc. cit.

[27] W. T. Edwards, Corrosion, ed. L. L. Shreir, (New York: John Wiley and Sons, 1963), p. 519.

[28] Remy, loc. cit.

[29] C. A. Hampel, The Encyclopedia of Electrochemistry (New York: Reinhold Publishing Corp., 1964), p. 416.

noteworthy similarity between the behavior of Nb and that of Ti, Zr, and Hf in HF.[30]

The minimum corrosion rates of the Fe - Nb alloys were observed to occur at about 1 - 1.5% by weight of Nb. This percentage coincides with the percentage where the $NbFe_2$ compound is no longer retained in the ferrite grains in solid solution, but begins to precipitate in the form of small grains in the grain boundaries and also in the ferrite matrix (see page 31). This coincidence may indicate the mechanism in which the Nb protects the alloy. A protective oxide film is probably formed on the alloy up to 1 - 1.5% by weight of Nb, and this causes the sharp decrease in corrosion rate. Another possible contributing factor to the decrease in corrosion rate is that the niobium may have also tied up the carbon impurity (.0035%) in the form of niobium carbide thus reducing the number of local cathodes and reducing their tendency to cause the iron to corrode as shown by Chappell[31] and Aitchison.[32] The precipitate of $NbFe_2$ and NbC is so fine that the particles actually cannot act as effective local cathodes because they may be quickly undermined by the corrosion process and are carried

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30 Straumanis and Chen, loc. cit.

31 Chappell, loc. cit.

32 Aitchison, loc. cit.

away, causing fluctuations in the rate. Above 1 - 1.5% Nb where the number and size of $NbFe_2$ grains increases sharply, local cell action probably out-weighs the effect of the protective film and causes the rise in corrosion rates. The normal potentials of Nb and Fe, being -1.1 and -.44 volts respectively, would give rise to local cells with the Fe acting as cathode and would not explain at all the protective action of small Nb amounts in Fe. It is assumed, as in the case of Cr, that Nb concentrations in Fe are covered with an adherent and acid resistive, surface oxide layer, possessing a more noble potential than that of Fe. This oxide layer may consist of a mixed Nb and Fe oxide. If the solid solubility limit is exceeded (1.5% Nb) $NbFe_2$ precipitates, as already mentioned, and acts cathodically toward the Fe and provides more and larger cathodic particles from which hydrogen can readily be evolved. This explanation is similar to that given by Hoar and Havenhand[33] for the fact that dead mild steels containing massive cementite are attacked more quickly than steels of equal carbon content but with more dispersed cementite.

The erratic behavior of the corrosion rate versus time curves for Fe and the Fe - Nb alloys must be

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[33] T. P. Hoar and D. Havenhand, "Factors Influencing the Rate of Attack of Mild Steels by Typical Weak Acid Media," J. Iron and Steel Institute, CXXXIII, (1936), p. 239p.

attributed to changing conditions on the metal surface. Changes on the metal surface include changes in thickness and nature of the protective oxide layers, dropout of $NbFe_2$, NbC and even Fe grains from the surface as corrosion proceeds. A minor influence may be the altering of the relative amounts of metal grains favorably oriented for corrosion and those unfavorably oriented as described by Welch.[34] Another factor which would influence the corrosion rate is the fact that the samples were prepared by powder metallurgy and are not as homogeneous in macroscopic structure as a wrought material. This means that as pores are exposed the actual surface area of the sample increases and as corrosion proceeds to smooth out the pores the surface area decreases. Another influence is the fact that as most of the pores in the metal sample were gradually closed during sintering, any impurities in the metal congregated at these pore sites and could cause points which were easily corroded. As these points were exposed and dissolved the corrosion rate changed.

The major change in the acid solutions is an increase in the metal ion concentration with a corresponding

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[34] Gary E. Welch, "Dissolution Rates of Zone Refined Iron and Steel in Hydrochloric and Sulfuric Acids and in Sodium Chloride Solutions," (partially published Master's Thesis, The University of Missouri - Rolla, Rolla, Missouri, 1963), p. 21.

decrease in the hydrogen ion concentration as corrosion proceeds. Another changeable factor is the amount of agitation of the acid by the bubbling of the argon. It was impossible to maintain exactly the same flow rate throughout the length of the corrosion run, in fact, adjustments were necessary every day.

The formation of a metal skin on the corrosion samples is explained by the difference in carbon content of the skin and the core as discussed above on page 45.

V. CONCLUSIONS

The obvious intent of any research is to provide new knowledge which may be applied directly or indirectly to a human need. In this investigation we have found that an addition of 1 - 1.5% by weight Nb to pure Fe produces a decrease in its corrosion rate of a factor of about 3. Application of this knowledge will be restricted to fairly specialized fields where corrosion resistance in iron is desired. It must be remembered that these findings may not extend to steel, the chief alloy of iron, because in steel the Nb may be tied up as NbC since there is a significant amount of carbon present. The corrosion resistance of stainless steel is improved by the addition of Nb, but this is due to the formation of a random precipitate of NbC within the grains rather than a continuous pattern of CrC at the grain boundaries.[35] This prevention of sensitization is a completely different mechanism than that which we have discussed for Nb in Fe. It is still unknown whether NbC is cathodically more active than Fe. Therefore, applications may be limited to uses of iron where corrosion resistance will be helpful and a high purity and strength are not necessary.

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[35] Carl H. Samans, Metallic Materials in Engineering (New York: The MacMillan Company, 1963), p. 448.

VI. SUMMARY

The valence of niobium ions going into solution was determined by dissolving the metal in hot HF and measuring the volume of hydrogen evolved. The valence was determined to be +5 which agrees with values of Nb in the salt NbF_5 reported in the literature.

The corrosion rates of Nb, NbH_x, Fe and Fe - Nb alloys were determined in various concentrations of HCl, H_2SO_4, and HF. It was found that the Nb and NbH_x were very resistive to corrosion and were slowly attacked only by HF. The corrosion rates of iron containing 1 - 1.5% by weight of Nb were reduced on the average by a factor of 3 compared to pure iron under identical conditions.

It was found that a metal skin was produced on the surface of iron corrosion samples (obtained by powder metallurgy) if the carbon content of the surface layer was lower than that of the core of the sample. This condition was eliminated by reducing the surface oxides and decarburizing the iron powder in a hydrogen atmosphere before pressing the samples.

The electrochemical potentials of Nb, NbH_x, Fe and Fe - Nb alloys were determined in the same acids in which the corrosion studies were performed. It was found that the potentials of Nb and NbH_x became more negative with increasing concentration of the acids,

similar to the behavior of Ti, Hf, and Zr.[36] The potentials of Fe and Fe - Nb alloys, on the contrary, become more positive with increasing concentration of the acids in agreement with Evans.[37] There was also a nearly linear relation between the potentials and the weight percent Nb in the Fe - Nb alloys with the potential becoming more positive with increasing Nb content.

There are several areas of investigation which remain untouched in the present work. The first is the determination of the exact nature of the protective film on Nb. Also the nature of the mechanism due to which the resistance to corrosion of pure Fe increases upon addition of more than 1 - 1.5% by weight Nb should be investigated. The skin formation should be studied further and determinations made as to whether this phenomenon is peculiar to iron alone or to powder metallurgy samples alone. Some work can also be done to determine current densities of Fe - Nb alloys dissolving in acids.

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[36] Straumanis and Chen, loc. cit.

[37] Evans, loc. cit.

VII. APPENDIX

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LIST OF ILLUSTRATIONS FOR THE APPENDIX

NIOBIUM VALENCE DETERMINATION

RUN #1

26.87398 g.	weight of Nb and flask
-26.86841 g.	weight of flask
.00557 g.	Nb
- .00058 g.	Nb residue
.00499 g.	Nb dissolved

736.4	mm.Hg	observed
- 19.8	mm.Hg	H_2O vapor pressure correction
716.6		
- 2.8	mm.Hg	brass scale correction
713.8		
+ .3	mm.Hg	capillary depression correction
714.1	mm.Hg	corrected

START: 19.41°C in bath
0.90 ml. burette reading

END: 19.41°C in bath
4.40 ml. burette reading
736.4 mm.Hg barometric pressure
22.0°C room temperature

4.40 ml. end
- .90 ml. start
3.50

273.2°
+ 22.0°
295.2°K room temperature

$$\frac{(3.50 \text{ ml. } H_2)(714.1 \text{ mm. Hg})}{295.2°K} = X \frac{760 \text{ mm. Hg.}}{273.2°K}$$

$$X = (3.50 \text{ ml. } H_2) \frac{714.1}{760} \frac{273.2}{295.2}$$

$$X = 3.50(.9396)(.9255) = 3.04 \text{ ml. } H_2 \text{ corrected}$$

$$v = \frac{(3.04)(92.906)}{(.00499)(11,207)} = \underline{\underline{5.050}}$$

NIOBIUM VALENCE DETERMINATION

RUN #2

27.58532 g.	weight of Nb and flask
-27.57497 g.	weight of flask
.01035 g.	Nb
- .00168 g.	Nb residue
.00867 g.	Nb dissolved

737.8	mm.Hg	observed
- 20.1	mm.Hg	H_2O vapor pressure correction
717.7		
- 2.6	mm.Hg	brass scale correction
715.1		
+ .3	mm.Hg	capillary depression correction
715.4	mm.Hg	corrected

START: 19.72°C in bath
0.60 ml. burette reading

END: 19.72°C in bath
6.51 ml. burette reading
22.2°C room temperature
737.8 mm.Hg barometric pressure

6.51 ml. end
- .60 ml. start
5.91

273.2°
+ 22.2°
295.4°K room temperature

$$\frac{(5.91 \text{ ml. } H_2)(715.4 \text{ mm. Hg})}{295.4^\circ K} = X \frac{760 \text{ mm. Hg}}{273.2^\circ K}$$

$$X = (5.91) \frac{715.4}{760} \frac{273.2}{295.4}$$

$$X = 5.91(.9413)(.9248) = 5.14 \text{ mm. } H_2 \text{ corrected}$$

$$v = \frac{(5.14)(92.906)}{(.00867)(11,207)} = \underline{\underline{4.915}}$$

NIOBIUM VALENCE DETERMINATION

RUN #3

34.29368 g.	weight of Nb and flask
-34.28287 g.	weight of flask
.01081 g.	Nb
- .00208 g.	Nb residue
.00873 g.	Nb dissolved

737.3	mm.Hg	observed
- 20.4	mm.Hg	H_2O vapor pressure correction
716.9		
- 2.7	mm.Hg	brass scale correction
714.2		
+ .3	mm.Hg	capillary depression correction
714.5	mm.Hg	corrected

START: 19.38°C in bath
.68 ml. burette reading

END: 6.68 ml. burette reading
19.38°C in bath
22.2°C room temperature
737.3 mm.Hg barometric pressure

6.68 ml. end	273.2°
- .68 ml. start	+ 22.2°
6.00	295.4°

$$\frac{(6.00 \text{ ml. } H_2)(714.5 \text{ mm. Hg})}{295.4} = X \frac{760 \text{ mm. Hg}}{273.2}$$

$$X = (6.00) \frac{714.5}{760} \frac{273.2}{295.4}$$

$$X = 6.00(.9401)(.9248) = 5.22 \text{ mm. } H_2 \text{ corrected}$$

$$v = \frac{(5.22)(92.906)}{(.00873)(11,207)} = \underline{\underline{5.004}}$$

NIOBIUM VALENCE DETERMINATION

RUN #4

33.79508 g.	weight of Nb and flask
-33.78760 g.	weight of flask
.00748 g.	Nb
- .00134 g.	Nb residue
.00614 g.	Nb dissolved

733.3	mm.Hg	observed
- 19.8	mm.Hg	H_2O vapor pressure correction
713.5		
- 2.7	mm.Hg	brass scale correction
710.8		
+ .3	mm.Hg	capillary depression correction
711.1	mm.Hg	corrected

START: 20.2°C in bath
1.01 ml. burette reading

END: 5.30 ml. burette reading
20.2°C in bath
733.3 mm.Hg barometric pressure
22.0°C room temperature

5.30 ml. end	273.2°
-1.01 ml. start	+ 22.0°
4.29	295.2

$$\frac{(4.29 \text{ ml. } H_2)(711.1 \text{ mm. Hg})}{295.2} = X \quad \frac{760 \text{ mm. Hg}}{273.2}$$

$$X = (4.29 \text{ ml. } H_2) \quad \frac{711.1}{760} \quad \frac{273.2}{295.2}$$

$$X = 4.29\ (.9357)(.9255) = 3.72 \text{ ml. } H_2 \text{ corrected}$$

$$v = \frac{(3.72)\ (92.906)}{(.00614)(11,207)} = \underline{5.023}$$

SAMPLE CALCULATIONS FOR DETERMINING CORROSION RATES

$$\text{CORROSION RATE} = \frac{\text{WEIGHT LOSS}}{(\text{SURFACE AREA})(\text{TIME})}$$

Sample data taken from the corrosion of 1%Nb - 99% Fe during the first 24 hours in 1N H_2SO_4.

Total weight loss of two 1%Nb samples in 1N H_2SO_4:

$$.0066g + .0048g = .0114g \text{ or } 11.4mg$$

Total surface area of two 1%Nb samples in 1N H_2SO_4:

$$4.052\ cm^2 + 3.897\ cm^2 = 7.949\ cm^2$$

$$\text{CORROSION RATE} = \frac{11.4mg}{(7.949\ cm^2)(24\ hrs)}$$

$$\text{CORROSION RATE} = .060mg\ cm^{-2}\ hr^{-1}$$

TABLE 1

THE CORROSION RATES OF NIOBIUM IN HF

TIME (hours)	CORROSION RATES ($mg\ cm^{-2}\ hr^{-1}$) 1N HF	1N HF	5N HF	10N HF
24	.017	.030	.055	.090
48	.005	.011	.015	.038
72	.003	.001	.016	.022
96	.015	.020	.038	.063
120	.005	.023	.011	.036
144	.030	.042	.044	.077
168		.016	.000	.026
192		.017	.025	.069
216		.030	.033	.079
240		.005	.025	.071
264		.015	.046	.103
288		.013	.042	.088
312		.016	.056	.099
336		.009	.031	.075
360		.020	.062	.117
Average Maximum Corrosion Rate	.013	.018	.033	.070

TABLE 2

THE CORROSION RATES OF NbH_x in HF

TIME (hours)	CORROSION RATES ($mg\ cm^{-2}\ hr^{-1}$) .1N HF	1N HF	2N HF
96	.001	.006	.010
120	.007	.011	.019
144	.002	.009	.013
168	.005	.015	.020
264	.003		
288	.006		
Average Maximum Corrosion Rate	.004	.010	.016

TABLE 3

THE CORROSION RATES OF IRON IN HCl

TIME (hours)	CORROSION RATES ($mg\ cm^{-2}\ hr^{-1}$) 1N HCl	2N HCl	4N HCl
48	.151	.237	.975
72	.245	.512	2.247
96	.342	.742	2.276
144	.297	.553	1.316
168	.398	1.177	1.733
216	.431	1.226	1.233
240	.404	.711	.539
264	.331	.754	.579
Average Maximum Corrosion Rate	.325	.739	1.362

TABLE 4

THE CORROSION RATES OF IRON IN HF

TIME (hours)	CORROSION RATES (mg cm^{-2} hr^{-1}) 1N HF	2N HF	5N HF	10N HF
24	0.677	1.418	5.319	5.533
48	1.502	2.248	2.657	2.772
72	0.603	1.223	.295	.579
96	.043	0.151	.176	.080
120	.015	0.021	1.633	1.706
144			.380	.329
168			.094	.110
192			.259	.654
216			.263	.519
240			.236	.523
264			.137	.535
Average Maximum Corrosion Rate	.568	1.012	1.041	1.213

TABLE 5

THE CORROSION RATES OF IRON IN H_2SO_4

TIME (hours)	CORROSION RATES (mg cm^{-2} hr^{-1}) .5N H_2SO_4	1N H_2SO_4	2N H_2SO_4
48	.923	.483	1.606
72	1.204	.873	2.238
96	1.536	1.305	2.094
144	1.166	.979	1.269
168	.400	1.325	1.912
216		1.399	1.704
240		.697	.689
262		.742	.864
Average Maximum Corrosion Rate	1.046	.975	1.547

TABLE 6

THE CORROSION RATES OF IRON-NIOBIUM ALLOYS IN 1N HCl

TIME (hours)	CORROSION RATES ($mg\ cm^{-2}\ hr^{-1}$) 1%Nb	2%Nb	3%Nb	4%Nb	5%Nb
24	.014	.011	.017	.011	.007
48	.021	.016	.009	.025	.016
72	.015	.005	.010	.037	.024
96	.023	.008	.010	.040	.034
120	.019	.000	.000	.024	.033
144	.021	.011	.014	.034	.038
168	.015	.000	.000	.018	.033
288	.023	.006	.007	.040	.084
312	.037	.009	.010	.045	.066
336	.027	.003	.003	.040	.030
360	.024	.003	.002	.037	.028
384	.030	.008	.008	.046	.034
408	.029	.008	.012	.047	.028
432	.023	.000	.000	.043	.017
Average Maximum Corrosion Rate	.023	.006	.007	.035	.034

TABLE 7

THE CORROSION RATES OF IRON-NIOBIUM ALLOYS IN 1N HF

TIME (hours)	CORROSION RATES ($mg\ cm^{-2}\ hr^{-1}$) 1%Nb	2%Nb	3%Nb	4%Nb	5%Nb
24	.002	.010	.203	.256	.101
48	.014	.026	.268	.294	.147
72	.014	.032	.346	.395	.303
96	.018	.054			.403
120	.007	.053			.427
144	.015	.056			.282
168	.006	.048			.232
288	.013	.074			.023
312	.013	.086			
336	.012	.080			
360	.014	.074			
384	.016	.084			
408	.021	.078			
432	.006	.084			
Average Maximum Corrosion Rate	.012	.060	.272	.315	.240

TABLE 8

THE CORROSION RATES OF IRON-NIOBIUM ALLOYS IN 1N H_2SO_4

TIME (hours)	CORROSION RATES ($mg\ cm^{-2}\ hr^{-1}$) 1%Nb	2%Nb	3%Nb	4%Nb	5%Nb
24	.060	.064	.060	.081	.088
48	.035	.043	.043	.052	.058
72	.050	.057	.061	.089	.078
96	.051	.063	.063	.109	.094
120	.052	.066	.067	.111	.090
144	.045	.055	.052	.074	.073
168	.040	.050	.040	.071	.058
288	.047	.070	.067	.156	.114
312	.028	.034	.035	.086	.092
336	.032	.038	.041	.098	.079
360	.025	.032	.033	.097	.084
384	.037	.047	.047	.119	.104
408	.030	.050	.048	.109	.099
432	.028	.032	.038	.124	.125
Average Maximum Corrosion Rate	.040	.050	.050	.098	.088

TABLE 9

THE CORROSION RATES OF .5% NIOBIUM - 99.5% IRON IN 1 AND 2N HCl, H_2SO_4, AND HF

TIME (hours)	CORROSION RATES ($mg\ cm^{-2}\ hr^{-1}$) 1N HCl	2N HCl	1N H_2SO_4	2N H_2SO_4	1N HF	2N HF
24	.054	.115	.182	.127	.365	.406
48	.033	.086	.233	.146	.323	.399
72	.041	.115	.285	.173	.180	.270
96	.041	.131	.295	.177	.074	.120
120	.040	.159	.323	.233	.052	.070
144	.044	.196	.353	.258	.035	.053
168	.044	.220	.290	.205	.024	.040
192	.050	.305	.324	.243	.045	.058
216	.063	.333	.280	.227	.032	.058
240	.064	.386	.301	.219	.015	.040
264	.080	.427	.320	.243	.034	.051
288	.069	.486	.336	.252	.030	.047
312	.063	.477	.314	.226	.016	.026
336	.058	.439	.233	.211	.010	.025
360	.091	.469	.307	.278	.033	.036
Average Maximum Corrosion Rate	.056	.290	.292	.215	.085	.113

SAMPLE CALCULATIONS FOR POTENTIAL DETERMINATIONS

Potential on normal hydrogen scale = Potential on calomel scale + Potential of calomel electrode on normal hydrogen scale

Sample data taken from potential of iron in 1N H_2SO_4 at ten minutes.

-.5683v.	potentiometer reading
+.2750v.	potential of calibrated calomel electrode
-.2933v.	potential on normal hydrogen scale

TABLE 10

THE ELECTROCHEMICAL POTENTIAL OF Fe IN 1N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2651	90	-.2979
1	-.2710	100	-.2979
2	-.2760	110	-.2979
3	-.2779	120	-.2979
4	-.2791	130	-.2979
5	-.2799	140	-.2979
6	-.2804	150	-.2979
7	-.2810		
8	-.2812		
9	-.2820		
10	-.2826		
15	-.2848		
20	-.2849		
25	-.2870		
30	-.2896		
40	-.2920		
50	-.2949		
60	-.2956		
70	-.2968		
80	-.2979		

TABLE 11

THE ELECTROCHEMICAL POTENTIAL OF .5%Nb-99.5%Fe IN 1N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2381	90	-.2698
1	-.2411	100	-.2711
2	-.2420	110	-.2732
3	-.2422	120	-.2750
4	-.2425	130	-.2768
5	-.2431	140	-.2784
6	-.2440	150	-.2800
7	-.2448	160	-.2811
8	-.2451	170	-.2819
9	-.2459	180	-.2828
10	-.2464	190	-.2831
15	-.2484	200	-.2834
20	-.2502	210	-.2840
25	-.2514	220	-.2848
30	-.2534	230	-.2848
40	-.2565	240	-.2848
50	-.2586	250	-.2848
60	-.2621	260	-.2848
70	-.2664		
80	-.2690		

TABLE 12

THE ELECTROCHEMICAL POTENTIAL OF 1%Nb-99%Fe IN 1N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2431	90	-.2459
1	-.2508	100	-.2450
2	-.2530	110	-.2455
3	-.2535	120	-.2458
4	-.2536	130	-.2469
5	-.2537	140	-.2472
6	-.2541	150	-.2489
7	-.2538	160	-.2498
8	-.2538	170	-.2504
9	-.2538	180	-.2507
10	-.2538	190	-.2509
15	-.2530	200	-.2520
20	-.2524	210	-.2526
25	-.2519	220	-.2526
30	-.2510	270	-.2539
40	-.2508	280	-.2541
50	-.2499	290	-.2541
60	-.2487	300	-.2541
70	-.2478	310	-.2541
80	-.2461	320	-.2541

TABLE 13

THE ELECTROCHEMICAL POTENTIAL OF 2%Nb-98%Fe IN 1N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2290	100	-.2382
1	-.2370	110	-.2399
2	-.2339	120	-.2418
3	-.2331	130	-.2427
4	-.2333	140	-.2431
5	-.2335	150	-.2436
6	-.2336	160	-.2441
7	-.2336	170	-.2450
8	-.2334	180	-.2455
9	-.2334	190	-.2457
10	-.2334	200	-.2459
15	-.2330	210	-.2461
20	-.2331	220	-.2464
25	-.2332	230	-.2468
30	-.2338	240	-.2470
40	-.2345	250	-.2470
50	-.2354	260	-.2470
60	-.2358	270	-.2470
70	-.2361		
90	-.2373		

TABLE 14

THE ELECTROCHEMICAL POTENTIAL OF 3%Nb-97%Fe IN 1N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2400	90	-.2339
1	-.2360	100	-.2331
2	-.2320	110	-.2338
3	-.2295	120	-.2347
4	-.2280	130	-.2358
5	-.2270	140	-.2369
6	-.2264	150	-.2376
7	-.2269	160	-.2381
8	-.2256	170	-.2388
9	-.2260	180	-.2388
10	-.2260	190	-.2388
15	-.2257	200	-.2388
20	-.2258	210	-.2388
25	-.2260		
30	-.2269		
40	-.2280		
50	-.2300		
60	-.2310		
70	-.2320		
80	-.2331		

TABLE 15

THE ELECTROCHEMICAL POTENTIAL OF

4%Nb-96%Fe IN HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.1992	90	-.2129	300	-.2305
1	-.2021	100	-.2151	310	-.2310
2	-.2028	110	-.2160	320	-.2312
3	-.2028	120	-.2175	330	-.2315
4	-.2028	130	-.2180	340	-.2320
5	-.2022	140	-.2191	350	-.2320
6	-.2018	150	-.2200	360	-.2320
7	-.2010	160	-.2209	370	-.2320
8	-.2008	170	-.2215	380	-.2320
9	-.2000	180	-.2221		
10	-.2997	190	-.2238		
15	-.2000	200	-.2245		
20	-.2000	210	-.2255		
25	-.2014	220	-.2258		
30	-.2032	230	-.2265		
40	-.2055	240	-.2280		
50	-.2078	250	-.2281		
60	-.2098	260	-.2282		
70	-.2115	280	-.2293		
80	-.2125	290	-.2295		

TABLE 16

THE ELECTROCHEMICAL POTENTIAL OF

5%Nb-95%Fe IN 1N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2190	90	-.2140
1	-.2232	100	-.2162
2	-.2170	110	-.2180
3	-.2149	120	-.2195
4	-.2110	130	-.2210
5	-.2070	140	-.2230
6	-.2040	150	-.2244
7	-.2022	160	-.2255
8	-.2001	170	-.2265
9	-.1987	200	-.2275
10	-.1980	220	-.2296
15	-.1975	230	-.2309
20	-.1955	240	-.2309
25	-.1954	250	-.2309
30	-.1960	260	-.2309
40	-.1971	270	-.2309
50	-.2010		
60	-.2060		
70	-.2087		
80	-.2118		

TABLE 17

THE ELECTROCHEMICAL POTENTIAL OF Fe IN 2N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2580	90	-.2786
1	-.2710	100	-.2793
2	-.2720	110	-.2796
3	-.2739	120	-.2800
4	-.2740	130	-.2800
5	-.2741	140	-.2802
6	-.2745	150	-.2804
7	-.2746	160	-.2804
8	-.2747	170	-.2804
9	-.2749	180	-.2804
10	-.2750	190	-.2804
15	-.2750	200	-.2804
20	-.2751		
25	-.2755		
30	-.2756		
40	-.2760		
50	-.2761		
60	-.2766		
70	-.2775		
80	-.2780		

TABLE 18

THE ELECTROCHEMICAL POTENTIAL OF .5%Nb-99%Fe IN 2N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2368	90	-.2552	290	-.2620
1	-.2425	100	-.2560	300	-.2620
2	-.2441	110	-.2563	310	-.2620
3	-.2460	120	-.2569	320	-.2620
4	-.2468	130	-.2579	330	-.2620
5	-.2472	140	-.2580	340	-.2620
6	-.2481	150	-.2588	350	-.2620
7	-.2490	160	-.2590		
8	-.2499	170	-.2593		
9	-.2500	180	-.2597		
10	-.2500	190	-.2602		
15	-.2507	200	-.2607		
20	-.2518	210	-.2609		
25	-.2522	220	-.2610		
30	-.2527	230	-.2611		
40	-.2549	240	-.2612		
50	-.2569	250	-.2613		
60	-.2590	260	-.2615		
70	-.2521	270	-.2616		
80	-.2541	280	-.2616		

TABLE 19

THE ELECTROCHEMICAL POTENTIAL OF

1%Nb-99%Fe IN 2N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2233	90	-.2428	320	-.2334
1	-.2263	100	-.2419	330	-.2334
2	-.2326	110	-.2410	340	-.2334
3	-.2350	120	-.2401	350	-.2334
4	-.2356	130	-.2390		
5	-.2360	140	-.2370		
6	-.2366	150	-.2351		
7	-.2374	160	-.2340		
8	-.2388	170	-.2322		
9	-.2399	180	-.2321		
10	-.2402	190	-.2320		
15	-.2410	200	-.2320		
20	-.2412	220	-.2318		
25	-.2420	240	-.2320		
30	-.2423	250	-.2320		
40	-.2422	260	-.2322		
50	-.2431	270	-.2327		
60	-.2430	280	-.2330		
70	-.2425	300	-.2333		
80	-.2426	310	-.2334		

TABLE 20

THE ELECTROCHEMICAL POTENTIAL OF 2%Nb-98%Fe IN 2N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2030	90	-.2255	310	-.2250
1	-.2149	100	-.2258	320	-.2250
2	-.2185	110	-.2259	330	-.2250
3	-.2194	120	-.2267	340	-.2250
4	-.2200	130	-.2272		
5	-.2205	140	-.2284		
6	-.2209	150	-.2290		
7	-.2216	160	-.2290		
8	-.2220	170	-.2288		
9	-.2229	180	-.2285		
10	-.2234	190	-.2280		
15	-.2236	200	-.2269		
20	-.2241	210	-.2250		
25	-.2242	220	-.2245		
30	-.2245	230	-.2242		
40	-.2248	240	-.2241		
50	-.2250	270	-.2245		
60	-.2250	280	-.2248		
70	-.2251	290	-.2249		
80	-.2251	300	-.2250		

TABLE 21

THE ELECTROCHEMICAL POTENTIAL OF 3%Nb-97%Fe IN 2N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2323	90	-.2120
1	-.2270	100	-.2125
2	-.2248	110	-.2131
3	-.2235	120	-.2148
4	-.2232	130	-.2149
5	-.2230	140	-.2171
6	-.2230	150	-.2181
7	-.2230	160	-.2191
8	-.2228	170	-.2200
9	-.2222	180	-.2202
10	-.2220	190	-.2207
15	-.2200	200	-.2208
20	-.2181	210	-.2210
25	-.2170	220	-.2210
30	-.2160	230	-.2210
40	-.2151	240	-.2210
50	-.2142	250	-.2210
60	-.2129		
70	-.2125		
80	-.2121		

TABLE 22

THE ELECTROCHEMICAL POTENTIAL OF

4%Nb-96%Fe IN 2N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.1970	90	-.1975	290	-.2104
1	-.2005	100	-.1995	300	-.2104
2	-.2011	110	-.2010	310	-.2104
3	-.2010	120	-.2020	320	-.2104
4	-.2007	130	-.2024	330	-.2104
5	-.2003	140	-.2055	340	-.2104
6	-.2001	150	-.2062		
7	-.1998	160	-.2070		
8	-.1995	170	-.2075		
9	-.1990	180	-.2078		
10	-.1984	190	-.2082		
15	-.1978	200	-.2085		
20	-.1971	210	-.2089		
25	-.1970	220	-.2092		
30	-.1965	230	-.2094		
40	-.1964	240	-.2098		
50	-.1962	250	-.2099		
60	-.1967	260	-.2100		
70	-.1968	270	-.2102		
80	-.1970	280	-.2104		

TABLE 23

THE ELECTROCHEMICAL POTENTIAL OF 5%Nb-95%Fe IN 2N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2055	90	-.1922	300	-.2069
1	-.2138	100	-.1930	310	-.2071
2	-.2115	110	-.1943	320	-.2075
3	-.2088	120	-.1955	330	-.2075
4	-.2039	130	-.1962	340	-.2075
5	-.2020	140	-.1972	350	-.2075
6	-.2000	150	-.1980	360	-.2075
7	-.1980	170	-.2000	370	-.2075
8	-.1966	180	-.2010		
9	-.1955	190	-.2011		
10	-.1950	200	-.2018		
15	-.1938	210	-.2018		
20	-.1927	220	-.2019		
25	-.1924	230	-.2020		
30	-.1920	240	-.2026		
40	-.1914	250	-.2031		
50	-.1915	260	-.2040		
60	-.1915	270	-.2050		
70	-.1915	280	-.2055		
80	-.1920	290	-.2062		

TABLE 24

THE ELECTROCHEMICAL POTENTIAL OF Fe IN 4N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2305	90	-.2551
1	-.2340	100	-.2554
2	-.2352	110	-.2556
3	-.2360	120	-.2559
4	-.2364	130	-.2559
5	-.2371	140	-.2559
6	-.2382	150	-.2559
7	-.2390	160	-.2559
8	-.2396		
9	-.2400		
10	-.2406		
15	-.2435		
20	-.2449		
25	-.2475		
30	-.2500		
40	-.2515		
50	-.2515		
60	-.2524		
70	-.2530		
80	-.2545		

TABLE 25

THE ELECTROCHEMICAL POTENTIAL OF .5%Nb-99%Fe IN 4N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2014	90	-.2353	290	-.2419
1	-.2062	100	-.2360	300	-.2419
2	-.2093	110	-.2362	310	-.2419
3	-.2115	120	-.2371		
4	-.2128	130	-.2381		
5	-.2138	140	-.2392		
6	-.2148	150	-.2400		
7	-.2154	160	-.2405		
8	-.2159	170	-.2406		
9	-.2160	180	-.2405		
10	-.2160	190	-.2407		
15	-.2174	200	-.2408		
20	-.2208	210	-.2410		
25	-.2225	220	-.2410		
30	-.2235	230	-.2412		
40	-.2252	240	-.2412		
50	-.2286	250	-.2413		
60	-.2300	260	-.2415		
70	-.2321	270	-.2419		
80	-.2339	280	-.2419		

TABLE 26

THE ELECTROCHEMICAL POTENTIAL OF 1%Nb-99%Fe IN 4N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2090	90	-.2195
1	-.2120	100	-.2197
2	-.2131	110	-.2200
3	-.2141	120	-.2195
4	-.2142	130	-.2188
5	-.2150	140	-.2179
6	-.2158	150	-.2180
7	-.2163	160	-.2179
8	-.2170	170	-.2172
9	-.2171	180	-.2168
10	-.2171	190	-.2164
15	-.2175	200	-.2160
20	-.2177	210	-.2155
25	-.2179	220	-.2152
30	-.2181	230	-.2152
40	-.2195	240	-.2152
50	-.2215	250	-.2152
60	-.2219	260	-.2152
70	-.2218		
80	-.2201		

TABLE 27

THE ELECTROCHEMICAL POTENTIAL OF 2%Nb-98%Fe IN 4N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.1972	90	-.2135
1	-.2042	100	-.2121
2	-.2075	110	-.2111
3	-.2094	120	-.2102
4	-.2100	130	-.2093
5	-.2102	140	-.2088
6	-.2105	150	-.2080
7	-.2109	160	-.2072
8	-.2112	170	-.2065
9	-.2115	180	-.2062
10	-.2118	190	-.2061
15	-.2124	200	-.2055
20	-.2130	210	-.2048
25	-.2128	220	-.2045
30	-.2129	230	-.2045
40	-.2139	240	-.2045
50	-.2140	250	-.2045
60	-.2145	260	-.2045
70	-.2145		
80	-.2145		

TABLE 28

THE ELECTROCHEMICAL POTENTIAL OF 3%Nb-97%Fe IN 4N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2152	90	-.1940
1	-.2094	100	-.1939
2	-.2070	110	-.1941
3	-.2041	120	-.1943
4	-.2026	130	-.1945
5	-.2015	140	-.1936
6	-.2001	150	-.1930
7	-.1989	160	-.1920
8	-.1979	170	-.1916
9	-.1965	180	-.1921
10	-.1957	190	-.1935
15	-.1940	200	-.1943
20	-.1930	210	-.1943
25	-.1920	220	-.1948
30	-.1916	230	-.1952
40	-.1910	240	-.1960
50	-.1910	250	-.1962
60	-.1911	260	-.1962
70	-.1920	270	-.1962
80	-.1932	280	-.1962

TABLE 29

THE ELECTROCHEMICAL POTENTIAL OF 4%Nb-96%Fe IN 4N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.1944	90	-.1960	290	-.1905
1	-.1976	100	-.1857	300	-.1905
2	-.1989	110	-.1850	310	-.1905
3	-.1995	120	-.1848	320	-.1905
4	-.1993	130	-.1845		
5	-.1991	140	-.1845		
6	-.1986	150	-.1848		
7	-.1978	160	-.1851		
8	-.1969	170	-.1852		
9	-.1959	180	-.1857		
10	-.1951	190	-.1860		
15	-.1945	200	-.1864		
20	-.1940	210	-.1872		
25	-.1930	220	-.1882		
30	-.1920	230	-.1892		
40	-.1905	240	-.1896		
50	-.1895	250	-.1900		
60	-.1882	260	-.1902		
70	-.1875	270	-.1905		
80	-.1865	280	-.1905		

TABLE 30

THE ELECTROCHEMICAL POTENTIAL OF 5%Nb-95%Fe IN 4N HCl

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.1792	90	-.1778	300	-.1813
1	-.1864	100	-.1769	310	-.1818
2	-.1918	110	-.1753	320	-.1820
3	-.1921	120	-.1748	330	-.1820
4	-.1918	130	-.1739	340	-.1821
5	-.1917	140	-.1730	350	-.1822
6	-.1902	150	-.1732	360	-.1822
7	-.1891	160	-.1743	370	-.1822
8	-.1871	170	-.1745	380	-.1822
9	-.1848	190	-.1751	390	-.1822
10	-.1830	200	-.1759	400	-.1822
15	-.1794	210	-.1759	410	-.1822
20	-.1776	220	-.1764		
25	-.1765	230	-.1768		
30	-.1759	240	-.1778		
40	-.1751	250	-.1781		
50	-.1750	260	-.1783		
60	-.1752	270	-.1796		
70	-.1761	280	-.1803		
80	-.1969	290	-.1808		

TABLE 31

THE ELECTROCHEMICAL POTENTIAL OF

Fe IN .1N HF

TIME (min.)	POTENTIAL (volts)
0	-.3129
1	-.3204
2	-.3232
3	-.3266
4	-.3283
5	-.3298
6	-.3301
7	-.3309
8	-.3318
9	-.3331
10	-.3351
11	-.3352
12	-.3356
13	-.3356
14	-.3356
15	-.3356

TABLE 32

THE ELECTROCHEMICAL POTENTIAL OF

.5%Nb-99.5%Fe IN .1N HF

TIME (min.)	POTENTIAL (volts)
0	-.3210
1	-.3255
2	-.3280
3	-.3329
4	-.3342
5	-.3360
6	-.3365
7	-.3365
8	-.3365
9	-.3365

TABLE 33

THE ELECTROCHEMICAL POTENTIAL OF 1%Nb-99%Fe IN .1N HF

TIME (min.)	POTENTIAL (volts)
0	-.3165
1	-.3209
2	-.3240
3	-.3278
4	-.3296
5	-.3305
6	-.3318
7	-.3330
8	-.3337
9	-.3351
10	-.3359
11	-.3369
12	-.3369
13	-.3369
14	-.3369

TABLE 34

THE ELECTROCHEMICAL POTENTIAL OF 2%Nb-98%Fe IN .1N HF

TIME (min.)	POTENTIAL (volts)
0	-.3200
1	-.3251
2	-.3271
3	-.3304
4	-.3320
5	-.3320
6	-.3329
7	-.3336
8	-.3343
9	-.3351
10	-.3359
11	-.3365
12	-.3374
13	-.3375
14	-.3375
15	-.3375
16	-.3375

TABLE 35

THE ELECTROCHEMICAL POTENTIAL OF 3%Nb-97%Fe IN .1N HF

TIME (min.)	POTENTIAL (volts)
0	-.3283
1	-.3335
2	-.3348
3	-.3359
4	-.3368
5	-.3383
6	-.3392
7	-.3402
8	-.3405
9	-.3412
10	-.3413
11	-.3415
12	-.3419
13	-.3421
14	-.3421
15	-.3421
16	-.3421

TABLE 36

THE ELECTROCHEMICAL POTENTIAL OF

4%Nb-96%Fe IN .1N HF

TIME (min.)	POTENTIAL (volts)
0	-.3260
1	-.3280
2	-.3305
3	-.3310
4	-.3317
5	-.3323
6	-.3323
7	-.3331
8	-.3331
9	-.3331
10	-.3331

TABLE 37

THE ELECTROCHEMICAL POTENTIAL OF

5%Nb-95%Fe IN .1N HF

TIME (min.)	POTENTIAL (volts)
0	-.3250
1	-.3282
2	-.3305
3	-.3314
4	-.3314
5	-.3314
6	-.3314

TABLE 38

THE ELECTROCHEMICAL POTENTIAL OF Fe IN 1N HF

TIME (min.)	POTENTIAL (volts)
0	-.2959
1	-.3121
2	-.3181
3	-.3205
4	-.3223
5	-.3235
6	-.3243
7	-.3252
8	-.3259
9	-.3265
10	-.3268
11	-.3272
12	-.3275
13	-.3282
14	-.3283
15	-.3288
16	-.3288
17	-.3288
18	-.3288

TABLE 39

THE ELECTROCHEMICAL POTENTIAL OF .5%Nb-99.5%Fe IN 1N HF

TIME (min.)	POTENTIAL (volts)
0	-.3069
1	-.3150
2	-.3161
3	-.3195
4	-.3260
5	-.3274
6	-.3280
7	-.3298
8	-.3307
9	-.3311
10	-.3315
11	-.3320
12	-.3320
13	-.3320
14	-.3320

TABLE 40

THE ELECTROCHEMICAL POTENTIAL OF 1%Nb-99%Fe IN 1N HF

TIME (min.)	POTENTIAL (volts)
0	-.3060
1	-.3121
2	-.3192
3	-.3230
4	-.3240
5	-.3266
6	-.3282
7	-.3292
8	-.3305
9	-.3314
10	-.3319
11	-.3323
12	-.3323
13	-.3324
14	-.3327
15	-.3329
16	-.3329
17	-.3329
18	-.3329

TABLE 41

THE ELECTROCHEMICAL POTENTIAL OF 2%Nb-98%Fe IN 1N HF

TIME (min.)	POTENTIAL (volts)
0	-.2940
1	-.3060
2	-.3090
3	-.3128
4	-.3145
5	-.3154
6	-.3157
7	-.3166
8	-.3169
9	-.3173
10	-.3176
11	-.3179
12	-.3183
13	-.3184
14	-.3185
15	-.3187
16	-.3187
17	-.3187
18	-.3187
19	-.3187

TABLE 42

THE ELECTROCHEMICAL POTENTIAL OF 3%Nb-97%Fe IN 1N HF

TIME (min.)	POTENTIAL (volts)
0	-.2950
1	-.3069
2	-.3088
3	-.3094
4	-.3105
5	-.3110
6	-.3116
7	-.3119
8	-.3120
9	-.3122
10	-.3122
11	-.3122
12	-.3122

TABLE 43

THE ELECTROCHEMICAL POTENTIAL OF 4%Nb-96%Fe IN 1N HF

TIME (min.)	POTENTIAL (volts)
0	-.3069
1	-.3097
2	-.3123
3	-.3133
4	-.3140
5	-.3149
6	-.3151
7	-.3153
8	-.3153
9	-.3156
10	-.3159
11	-.3159
12	-.3159
13	-.3159

TABLE 44

THE ELECTROCHEMICAL POTENTIAL OF 5%Nb-95%Fe IN 1N HF

TIME (min.)	POTENTIAL (volts)
0	-.2934
1	-.3004
2	-.2992
3	-.2979
4	-.2961
5	-.2959
6	-.2942
7	-.2938
8	-.2931
9	-.2927
10	-.2921
11	-.2921
12	-.2921
13	-.2921

TABLE 45

THE ELECTROCHEMICAL POTENTIAL OF

Fe IN 2N HF

TIME (min.)	POTENTIAL (volts)
0	-.2964
1	-.2971
2	-.3009
3	-.3060
4	-.3109
5	-.3168
6	-.3181
7	-.3190
8	-.3191
9	-.3191
10	-.3191
11	-.3191

TABLE 46

THE ELECTROCHEMICAL POTENTIAL OF .5%Nb-99.5%Fe IN 2N HF

TIME (min.)	POTENTIAL (volts)
0	-.2956
1	-.3086
2	-.3132
3	-.3157
4	-.3162
5	-.3163
6	-.3173
7	-.3177
8	-.3180
9	-.3180
10	-.3180
11	-.3180

TABLE 47

THE ELECTROCHEMICAL POTENTIAL OF

1%Nb-99%Fe IN 2N HF

TIME (min.)	POTENTIAL (volts)
0	-.2943
1	-.3068
2	-.3090
3	-.3132
4	-.3137
5	-.3154
6	-.3165
7	-.3187
8	-.3187
9	-.3187
10	-.3187

TABLE 48

THE ELECTROCHEMICAL POTENTIAL OF

2%Nb-98%Fe IN 2N HF

TIME (min.)	POTENTIAL (volts)
0	-.2930
1	-.3130
2	-.3141
3	-.3146
4	-.3146
5	-.3146
6	-.3146

TABLE 49

THE ELECTROCHEMICAL POTENTIAL OF 3%Nb-97%Fe IN 2N HF

TIME (min.)	POTENTIAL (volts)
0	-.2887
1	-.3025
2	-.3035
3	-.3052
4	-.3040
5	-.3025
6	-.3020
7	-.3020
8	-.3020
9	-.3020

TABLE 50

THE ELECTROCHEMICAL POTENTIAL OF 4%Nb-96%Fe IN 2N HF

TIME (min.)	POTENTIAL (volts)
0	-.2862
1	-.3002
2	-.3062
3	-.3069
4	-.3054
5	-.3015
6	-.2990
7	-.2980
8	-.2977
9	-.2977
10	-.2977
11	-.2977

TABLE 51

THE ELECTROCHEMICAL POTENTIAL OF

5%Nb-95%Fe IN 2N HF

TIME (min.)	POTENTIAL (volts)
0	-.2870
1	-.3033
2	-.3044
3	-.2969
4	-.2958
5	-.2949
6	-.2949
7	-.2949
8	-.2949

TABLE 52

THE ELECTROCHEMICAL POTENTIAL OF Fe IN 5N HF

TIME (min.)	POTENTIAL (volts)
0	-.2721
1	-.2880
2	-.2911
3	-.2942
4	-.2953
5	-.2960
6	-.2964
7	-.2970
8	-.2974
9	-.2977
10	-.2980
11	-.2980
12	-.2981
13	-.2982
14	-.2982
15	-.2982
16	-.2982

TABLE 53

THE ELECTROCHEMICAL POTENTIAL OF .5%Nb-99.5%Fe IN 5N HF

TIME (min.)	POTENTIAL (volts)
0	-.2772
1	-.2841
2	-.2888
3	-.2907
4	-.2916
5	-.2930
6	-.2942
7	-.2956
8	-.2962
9	-.2967
10	-.2969
11	-.2969
12	-.2969
13	-.2969

TABLE 54

THE ELECTROCHEMICAL POTENTIAL OF

1%Nb-99%Fe IN 5N HF

TIME (min.)	POTENTIAL (volts)
0	-.2740
1	-.2790
2	-.2817
3	-.2822
4	-.2861
5	-.2868
6	-.2894
7	-.2917
8	-.2936
9	-.2942
10	-.2947
11	-.2950
12	-.2950
13	-.2950
14	-.2950

TABLE 55

THE ELECTROCHEMICAL POTENTIAL OF

2%Nb-98%Fe IN 5N HF

TIME (min.)	POTENTIAL (volts)
0	-.2820
1	-.2868
2	-.2856
3	-.2850
4	-.2846
5	-.2839
6	-.2829
7	-.2829
8	-.2829
9	-.2829

TABLE 56

THE ELECTROCHEMICAL POTENTIAL OF 3%Nb-97%Fe IN 5N HF

TIME (min.)	POTENTIAL (volts)
0	-.2671
1	-.2770
2	-.2825
3	-.2829
4	-.2817
5	-.2807
6	-.2792
7	-.2787
8	-.2776
9	-.2772
10	-.2772
11	-.2772
12	-.2772

TABLE 57

THE ELECTROCHEMICAL POTENTIAL OF 4%Nb-96%Fe IN 5N HF

TIME (min.)	POTENTIAL (volts)
0	-.2555
1	-.2693
2	-.2720
3	-.2739
4	-.2735
5	-.2742
6	-.2747
7	-.2735
8	-.2731
9	-.2711
10	-.2681
11	-.2675
12	-.2675
13	-.2675
14	-.2675

TABLE 58

THE ELECTROCHEMICAL POTENTIAL OF 5%Nb-95%Fe IN 5N HF

TIME (min.)	POTENTIAL (volts)
0	-.2459
1	-.2590
2	-.2543
3	-.2557
4	-.2578
5	-.2598
6	-.2613
7	-.2628
8	-.2640
9	-.2640
10	-.2640
11	-.2640

TABLE 59

THE ELECTROCHEMICAL POTENTIAL OF IRON IN .5N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2590	90	-.2960
1	-.2740	100	-.2959
2	-.2796	110	-.2959
3	-.2829	120	-.2959
4	-.2845	130	-.2959
5	-.2859	140	-.2959
6	-.2869	150	-.2959
7	-.2880	160	-.2959
8	-.2889		
9	-.2896		
10	-.2900		
15	-.2920		
20	-.2928		
25	-.2940		
30	-.2949		
40	-.2956		
50	-.2960		
60	-.2962		
70	-.2961		
80	-.2960		

TABLE 60

THE ELECTROCHEMICAL POTENTIAL OF .5%Nb-99.5%Fe IN .5N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2490	130	-.2932
1	-.2606	140	-.2935
2	-.2672	150	-.2940
3	-.2708	160	-.2942
4	-.2733	170	-.2946
5	-.2750	180	-.2948
6	-.2764	360	-.2942
8	-.2773	430	-.2935
9	-.2783	440	-.2932
10	-.2791	450	-.2931
15	-.2820	460	-.2930
20	-.2833	470	-.2930
25	-.2849	480	-.2930
30	-.2862	490	-.2928
40	-.2875	500	-.2928
50	-.2886	510	-.2928
60	-.2890	520	-.2928
70	-.2907	530	-.2928
110	-.2928		
120	-.2930		

TABLE 61

THE ELECTROCHEMICAL POTENTIAL OF

1%Nb-99%Fe IN .5N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2332	100	-.2778
1	-.2632	110	-.2778
2	-.2684	120	-.2778
3	-.2714	130	-.2780
4	-.2730	140	-.2784
5	-.2748	150	-.2786
6	-.2760	170	-.2786
7	-.2766	180	-.2786
8	-.2776	190	-.2786
9	-.2780	200	-.2786
10	-.2786	210	-.2786
15	-.2804		
20	-.2810		
25	-.2812		
30	-.2812		
40	-.2808		
50	-.2804		
60	-.2790		
70	-.2786		
80	-.2780		
90	-.2778		

TABLE 62

THE ELECTROCHEMICAL POTENTIAL OF

2%Nb-98%Fe IN .5N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2432	90	-.2666
1	-.2548	100	-.2660
2	-.2584	110	-.2656
3	-.2606	120	-.2650
4	-.2624	130	-.2644
5	-.2634	140	-.2640
6	-.2644	150	-.2636
7	-.2654	160	-.2634
8	-.2658	170	-.2632
9	-.2662	180	-.2630
10	-.2668	200	-.2626
15	-.2676	210	-.2616
20	-.2684	220	-.2616
25	-.2684	230	-.2616
30	-.2688	240	-.2614
40	-.2688	250	-.2614
50	-.2686	260	-.2614
60	-.2682	270	-.2614
70	-.2676		
80	-.2670		

TABLE 63

THE ELECTROCHEMICAL POTENTIAL OF 3%Nb-97%Fe IN .5N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2432	90	-.2726
1	-.2578	100	-.2720
2	-.2636	110	-.2712
3	-.2678	120	-.2690
4	-.2696	130	-.2678
5	-.2708	150	-.2660
6	-.2718	160	-.2648
7	-.2730	170	-.2644
8	-.2736	180	-.2636
9	-.2744	190	-.2630
10	-.2748	200	-.2626
15	-.2760	210	-.2618
20	-.2766	260	-.2596
25	-.2766	270	-.2596
30	-.2766	280	-.2596
40	-.2762	290	-.2596
50	-.2754	300	-.2596
60	-.2746		
70	-.2738		
80	-.2732		

TABLE 64

THE ELECTROCHEMICAL POTENTIAL OF 4%Nb-96%Fe IN .5N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2432	90	-.2596
1	-.2542	100	-.2596
2	-.2582	110	-.2590
3	-.2598	120	-.2590
4	-.2618	130	-.2590
5	-.2630	140	-.2590
6	-.2640	150	-.2592
7	-.2648	160	-.2592
8	-.2654	170	-.2592
9	-.2660	180	-.2594
10	-.2664	190	-.2596
15	-.2676	200	-.2596
20	-.2676	210	-.2596
25	-.2676	220	-.2596
30	-.2672	230	-.2596
40	-.2666	240	-.2596
50	-.2636		
60	-.2620		
70	-.2612		
80	-.2602		

TABLE 65

THE ELECTROCHEMICAL POTENTIAL OF 5%Nb-95%Fe IN .5N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2501	90	-.2565
1	-.2590	100	-.2565
2	-.2615	110	-.2566
3	-.2633	120	-.2566
4	-.2646	130	-.2567
5	-.2650	140	-.2569
6	-.2665	150	-.2572
7	-.2670	160	-.2572
8	-.2675	170	-.2572
9	-.2679	180	-.2573
10	-.2680	190	-.2576
15	-.2678	200	-.2576
20	-.2672	210	-.2576
25	-.2664	220	-.2576
30	-.2655		
40	-.2638		
50	-.2620		
60	-.2606		
70	-.2577		
80	-.2573		

TABLE 66

THE ELECTROCHEMICAL POTENTIAL OF IRON IN 1N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2600	90	-.2931
1	-.2758	100	-.2928
2	-.2831	110	-.2928
3	-.2872	120	-.2930
4	-.2891	130	-.2930
5	-.2902	140	-.2928
6	-.2913	160	-.2926
7	-.2920	170	-.2927
8	-.2926	180	-.2920
9	-.2928	190	-.2920
10	-.2933	200	-.2923
15	-.2940	210	-.2926
20	-.2942	220	-.2922
25	-.2942	230	-.2926
30	-.2940	240	-.2917
40	-.2940	440	-.2921
50	-.2938	450	-.2922
60	-.2935	460	-.2922
70	-.2932	470	-.2922
80	-.2928		

TABLE 67

THE ELECTROCHEMICAL POTENTIAL OF .5%Nb-99.5%Fe IN 1N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2400	90	-.2896
1	-.2576	100	-.2896
2	-.2650	110	-.2896
3	-.2692	120	-.2893
4	-.2723	140	-.2889
5	-.2742	150	-.2886
6	-.2761	160	-.2879
7	-.2780	170	-.2879
8	-.2793	180	-.2877
9	-.2805	190	-.2870
10	-.2817	200	-.2869
15	-.2848	250	-.2836
20	-.2863	260	-.2831
25	-.2877	270	-.2826
30	-.2877	280	-.2826
40	-.2883	290	-.2821
50	-.2883	300	-.2813
60	-.2891	310	-.2813
70	-.2896	320	-.2813
80	-.2892	330	-.2813

TABLE 68

THE ELECTROCHEMICAL POTENTIAL OF 1%Nb-99%Fe IN 1N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2450	100	-.2788
1	-.2598	110	-.2787
2	-.2641	120	-.2783
3	-.2678	130	-.2781
4	-.2700	140	-.2773
5	-.2713	150	-.2773
6	-.2734	160	-.2771
7	-.2747	170	-.2770
8	-.2753	190	-.2761
9	-.2763	200	-.2760
10	-.2770	210	-.2710
15	-.2790		
20	-.2799		
25	-.2804		
30	-.2804		
40	-.2804		
50	-.2804		
60	-.2808		
70	-.2804		
80	-.2799		
90	-.2797		

TABLE 69

THE ELECTROCHEMICAL POTENTIAL OF 2%Nb-98%Fe IN 1N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2380	90	-.2525
1	-.2546	100	-.2525
2	-.2618	110	-.2523
3	-.2635	120	-.2520
4	-.2650	310	-.2499
5	-.2655	320	-.2499
6	-.2660	330	-.2499
7	-.2666	340	-.2499
8	-.2665	350	-.2499
9	-.2662	360	-.2499
10	-.2659	370	-.2499
15	-.2632		
20	-.2602		
25	-.2585		
30	-.2580		
40	-.2560		
50	-.2547		
60	-.2542		
70	-.2538		
80	-.2530		

TABLE 70

THE ELECTROCHEMICAL POTENTIAL OF

3%Nb-97%Fe IN 1N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2450	90	-.2460	360	-.2382
1	-.2593	100	-.2432	370	-.2371
2	-.2612	110	-.2432	380	-.2377
3	-.2616	120	-.2429	390	-.2371
4	-.2612	130	-.2429	400	-.2369
5	-.2608	140	-.2416	410	-.2368
6	-.2595	150	-.2420	420	-.2368
7	-.2585	160	-.2420	430	-.2371
8	-.2571	170	-.2411	440	-.2370
9	-.2562	180	-.2415	450	-.2370
10	-.2559	190	-.2410		
15	-.2539	200	-.2409		
20	-.2522	210	-.2409		
25	-.2511	220	-.2405		
30	-.2507	230	-.2401		
40	-.2482	240	-.2401		
50	-.2477	250	-.2397		
60	-.2477	330	-.2390		
70	-.2477	340	-.2371		
80	-.2462	350	-.2378		

TABLE 71

THE ELECTROCHEMICAL POTENTIAL OF 4%Nb-96%Fe IN 1N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2459	90	-.2363
1	-.2510	100	-.2369
2	-.2522	110	-.2370
3	-.2529	120	-.2370
4	-.2532	130	-.2371
5	-.2523	140	-.2379
6	-.2510	150	-.2380
7	-.2472	160	-.2382
8	-.2449	170	-.2385
9	-.2434	180	-.2390
10	-.2422	190	-.2388
15	-.2396	200	-.2390
20	-.2381	210	-.2389
25	-.2390	220	-.2388
30	-.2371	230	-.2388
40	-.2370	240	-.2388
50	-.2362	250	-.2388
60	-.2361	260	-.2388
70	-.2356	270	-.2388
80	-.2361	280	-.2388

TABLE 72

THE ELECTROCHEMICAL POTENTIAL OF 5%Nb-95%Fe IN 1N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2390	90	-.2342	300	-.2345
1	-.2439	100	-.2350	310	-.2345
2	-.2460	110	-.2349	320	-.2345
3	-.2464	120	-.2349		
4	-.2474	130	-.2348		
5	-.2473	140	-.2345		
6	-.2470	150	-.2345		
7	-.2467	170	-.2345		
8	-.2462	180	-.2340		
9	-.2456	190	-.2341		
10	-.2450	200	-.2339		
15	-.2425	210	-.2333		
20	-.2402	220	-.2334		
25	-.2388	230	-.2335		
30	-.2378	240	-.2329		
40	-.2362	250	-.2330		
50	-.2363	260	-.2344		
60	-.2361	270	-.2345		
70	-.2362	280	-.2345		
80	-.2363	290	-.2345		

TABLE 73

THE ELECTROCHEMICAL POTENTIAL OF Fe IN 2N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2560	90	-.2737
1	-.2630	100	-.2737
2	-.2675		
3	-.2702		
4	-.2722		
5	-.2729		
6	-.2732		
7	-.2734		
8	-.2737		
9	-.2738		
10	-.2740		
15	-.2741		
20	-.2738		
25	-.2738		
30	-.2738		
40	-.2737		
50	-.2737		
60	-.2737		
70	-.2737		
80	-.2737		

TABLE 74

THE ELECTROCHEMICAL POTENTIAL OF .5%Nb-99%Fe IN 2N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2530	90	-.2708	300	-.2638
1	-.2575	100	-.2702	310	-.2634
2	-.2650	110	-.2698	320	-.2632
3	-.2688	120	-.2695	330	-.2629
4	-.2697	130	-.2691	340	-.2626
5	-.2705	140	-.2685	350	-.2625
6	-.2714	150	-.2680	360	-.2620
7	-.2719	160	-.2679	370	-.2615
8	-.2725	170	-.2675	380	-.2610
9	-.2725	180	-.2672	390	-.2610
10	-.2736	190	-.2669	400	-.2610
15	-.2735	200	-.2665	410	-.2610
20	-.2735	220	-.2659	420	-.2610
25	-.2735	230	-.2653		
30	-.2735	240	-.2652		
40	-.2732	250	-.2651		
50	-.2726	260	-.2648		
60	-.2721	270	-.2643		
70	-.2718	280	-.2641		
80	-.2712	290	-.2639		

TABLE 75

THE ELECTROCHEMICAL POTENTIAL OF 1%Nb-99%Fe IN 2N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2414	290	-.2498
1	-.2516	300	-.2492
2	-.2565	360	-.2485
3	-.2603	370	-.2485
4	-.2624	380	-.2485
5	-.2642	390	-.2485
6	-.2648		
7	-.2654		
8	-.2660		
9	-.2645		
10	-.2638		
15	-.2629		
20	-.2630		
25	-.2624		
30	-.2612		
140	-.2575		
150	-.2569		
160	-.2580		
270	-.2507		
280	-.2500		

TABLE 76

THE ELECTROCHEMICAL POTENTIAL OF 2%Nb-98%Fe IN 2N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2521	190	-.2530
1	-.2672	300	-.2509
2	-.2688	310	-.2501
3	-.2682	320	-.2500
4	-.2681	330	-.2495
5	-.2681	340	-.2489
6	-.2680	350	-.2495
7	-.2685	360	-.2495
8	-.2670	370	-.2500
9	-.2665	380	-.2505
10	-.2660	390	-.2501
15	-.2631	400	-.2502
20	-.2615	410	-.2505
25	-.2610	420	-.2505
30	-.2610	430	-.2505
40	-.2611	440	-.2505
50	-.2608		
60	-.2608		
170	-.2535		
180	-.2546		

TABLE 77

THE ELECTROCHEMICAL POTENTIAL OF 3%Nb-97%Fe IN 2N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2345	90	-.2419	300	-.2301
1	-.2490	100	-.2410	310	-.2295
2	-.2520	110	-.2408	320	-.2290
3	-.2530	120	-.2394	330	-.2283
4	-.2535	130	-.2388	340	-.2280
5	-.2538	140	-.2380	350	-.2270
6	-.2535	160	-.2360	360	-.2262
7	-.2530	170	-.2353	370	-.2256
8	-.2530	180	-.2352	380	-.2250
9	-.2526	190	-.2350	390	-.2244
10	-.2520	200	-.2342	400	-.2239
15	-.2500	210	-.2342	410	-.2238
20	-.2481	220	-.2339	420	-.2238
25	-.2478	230	-.2330	430	-.2238
30	-.2470	240	-.2330	440	-.2238
40	-.2462	250	-.2325		
50	-.2453	260	-.2323		
60	-.2449	270	-.2320		
70	-.2436	280	-.2314		
80	-.2430	290	-.2308		

TABLE 78

THE ELECTROCHEMICAL POTENTIAL OF 4%Nb-96%Fe IN 2N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2273	90	-.2246
1	-.2354	100	-.2244
2	-.2385	110	-.2243
3	-.2386	150	-.2235
4	-.2392	160	-.2226
5	-.2395	200	-.2205
6	-.2400	240	-.2182
7	-.2392	250	-.2180
8	-.2394	260	-.2170
9	-.2381	270	-.2168
10	-.2381	280	-.2165
15	-.2346	290	-.2163
20	-.2324	300	-.2163
25	-.2311	310	-.2163
30	-.2294	320	-.2163
40	-.2270		
50	-.2260		
60	-.2254		
70	-.2252		
80	-.2252		

TABLE 79

THE ELECTROCHEMICAL POTENTIAL OF 5%Nb-95%Fe IN 2N H_2SO_4

TIME (min.)	POTENTIAL (volts)	TIME (min.)	POTENTIAL (volts)
0	-.2083	100	-.2140
1	-.2110	120	-.2132
2	-.2116	140	-.2125
3	-.2124	200	-.2130
4	-.2131	210	-.2131
5	-.2140	220	-.2131
6	-.2146	230	-.2130
7	-.2146	240	-.2130
8	-.2150	250	-.2129
9	-.2152	260	-.2128
10	-.2150	270	-.2122
15	-.2163	280	-.2119
20	-.2160	290	-.2118
25	-.2163	300	-.2117
30	-.2165	310	-.2113
40	-.2155	320	-.2112
50	-.2139	330	-.2112
60	-.2140	340	-.2112
70	-.2137	350	-.2112
80	-.2138		

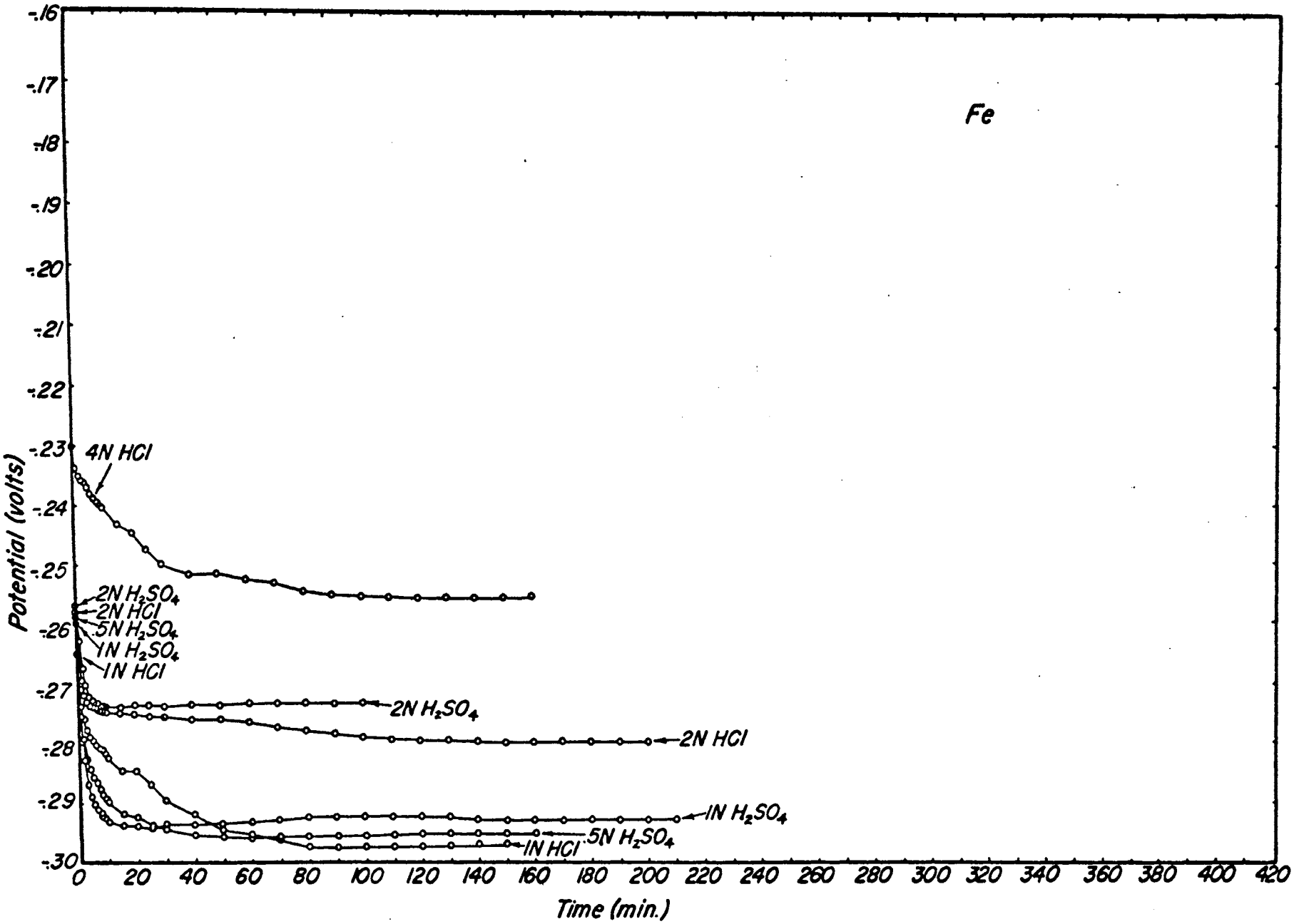

FIGURE 1
POTENTIAL VS. TIME FOR IRON IN HCl AND H_2SO_4

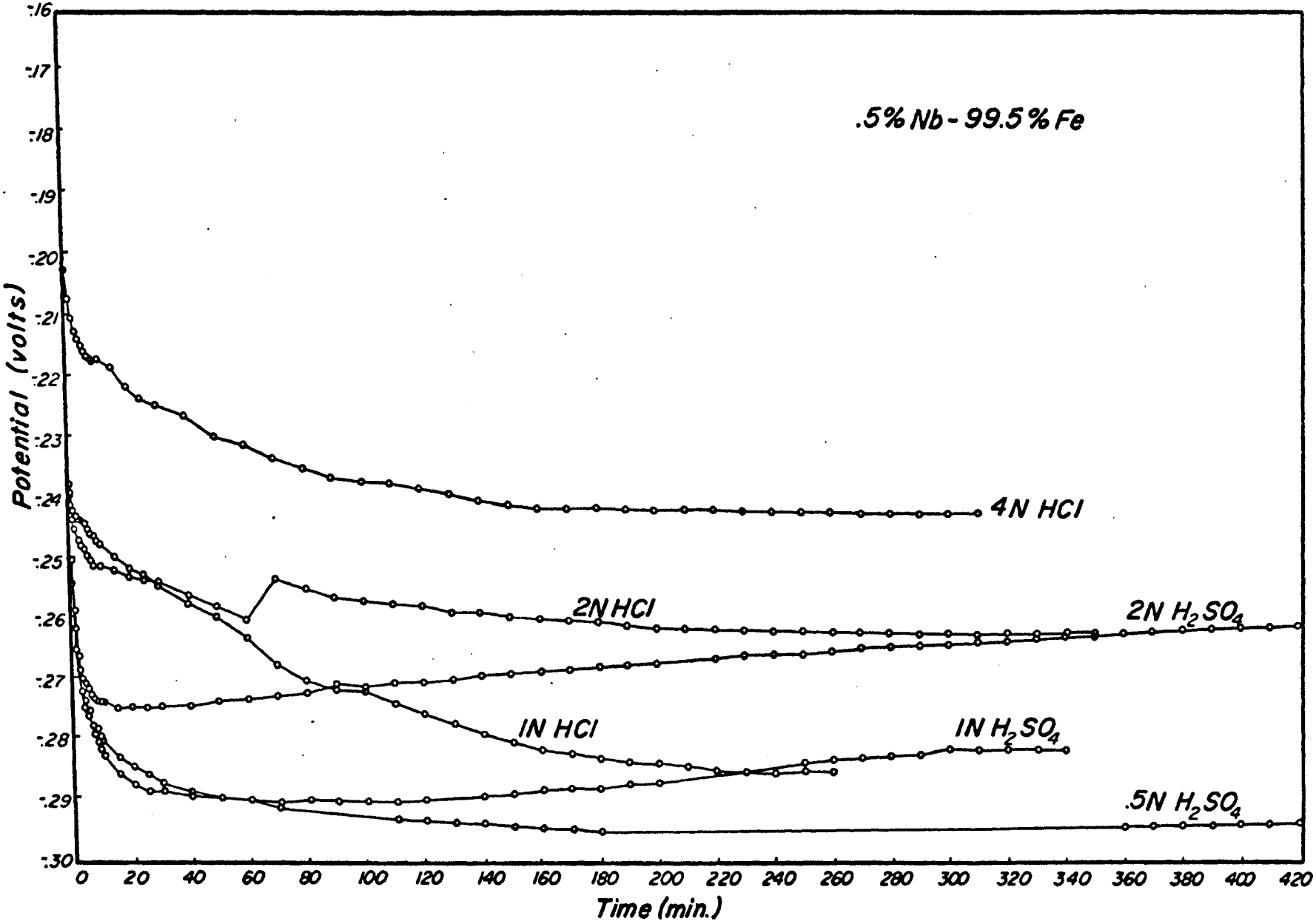

FIGURE 2
POTENTIAL VS. TIME FOR .5%Nb-99.5%Fe IN HCl AND H_2SO_4

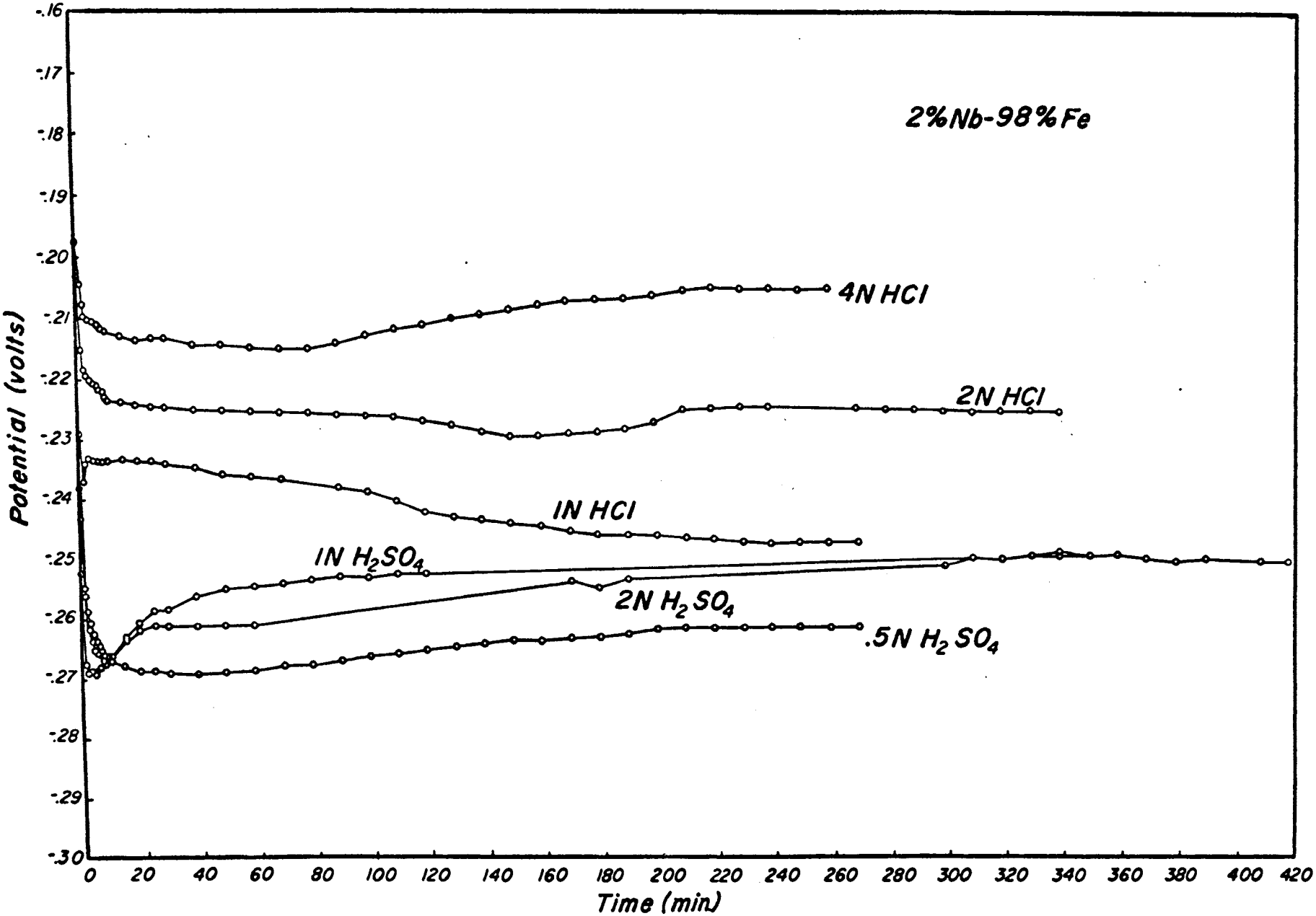

FIGURE 3
POTENTIAL VS. TIME FOR 2%Nb-98%Fe IN HCl AND H_2SO_4

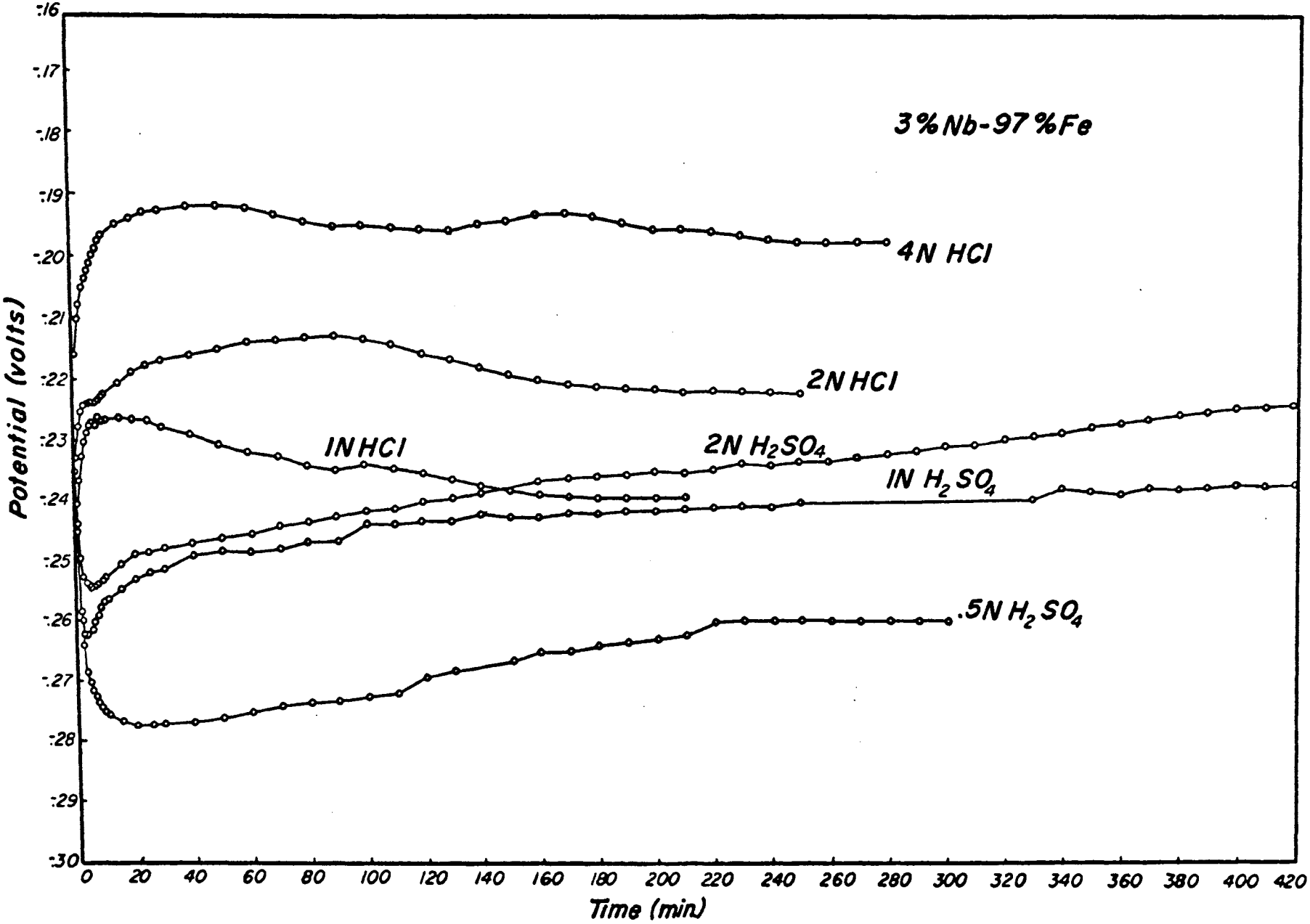

FIGURE 4
POTENTIAL VS. TIME FOR 3%Nb-97%Fe IN HCl AND H_2SO_4

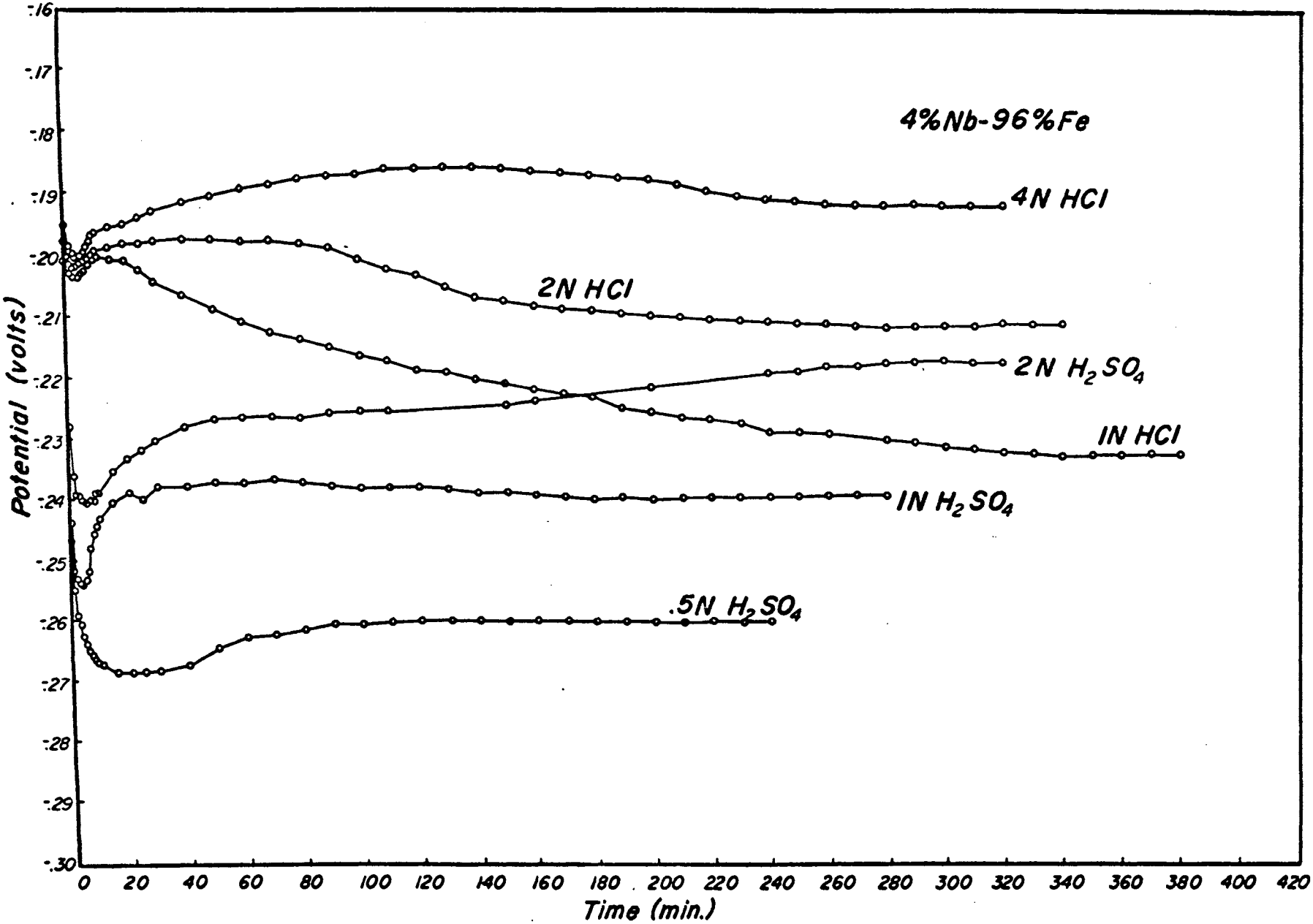

FIGURE 5
POTENTIAL VS. TIME FOR 4%Nb-96%Fe IN HCl AND H_2SO_4

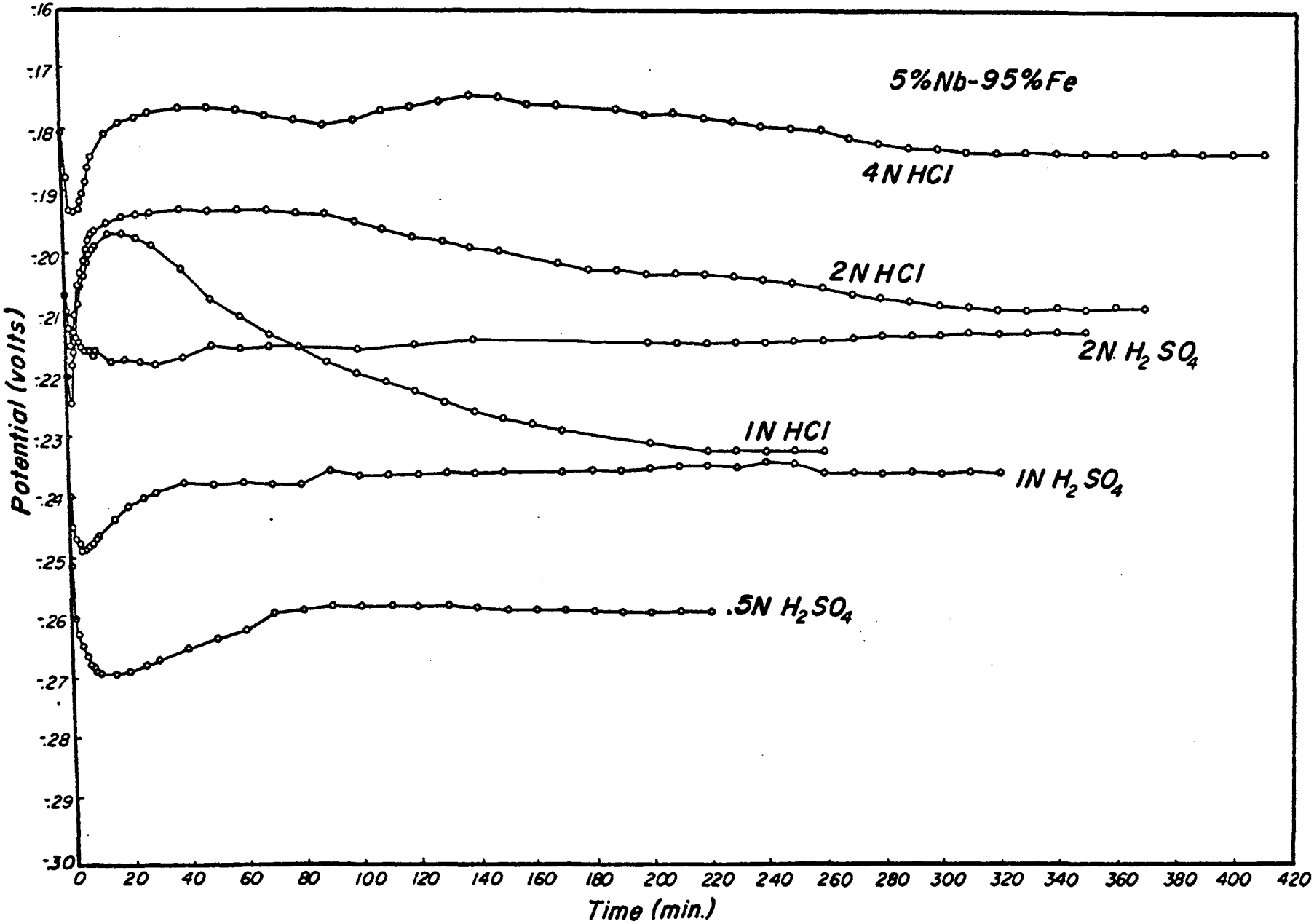

FIGURE 6
POTENTIAL VS. TIME FOR 5%Nb-95%Fe IN HCl AND H_2SO_4

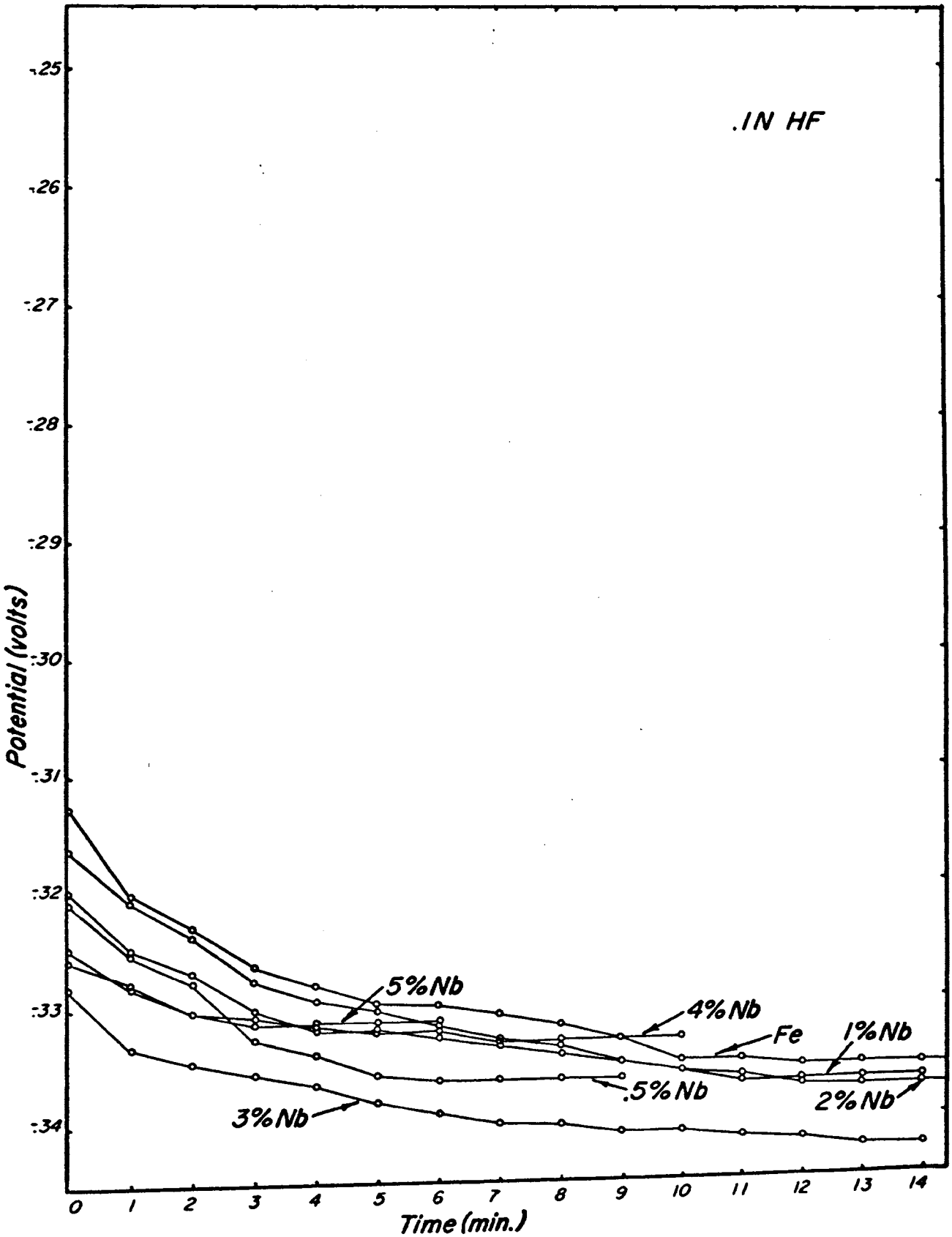

FIGURE 7

POTENTIAL VS. TIME FOR IRON AND IRON-NIOBIUM ALLOYS IN .1N HF

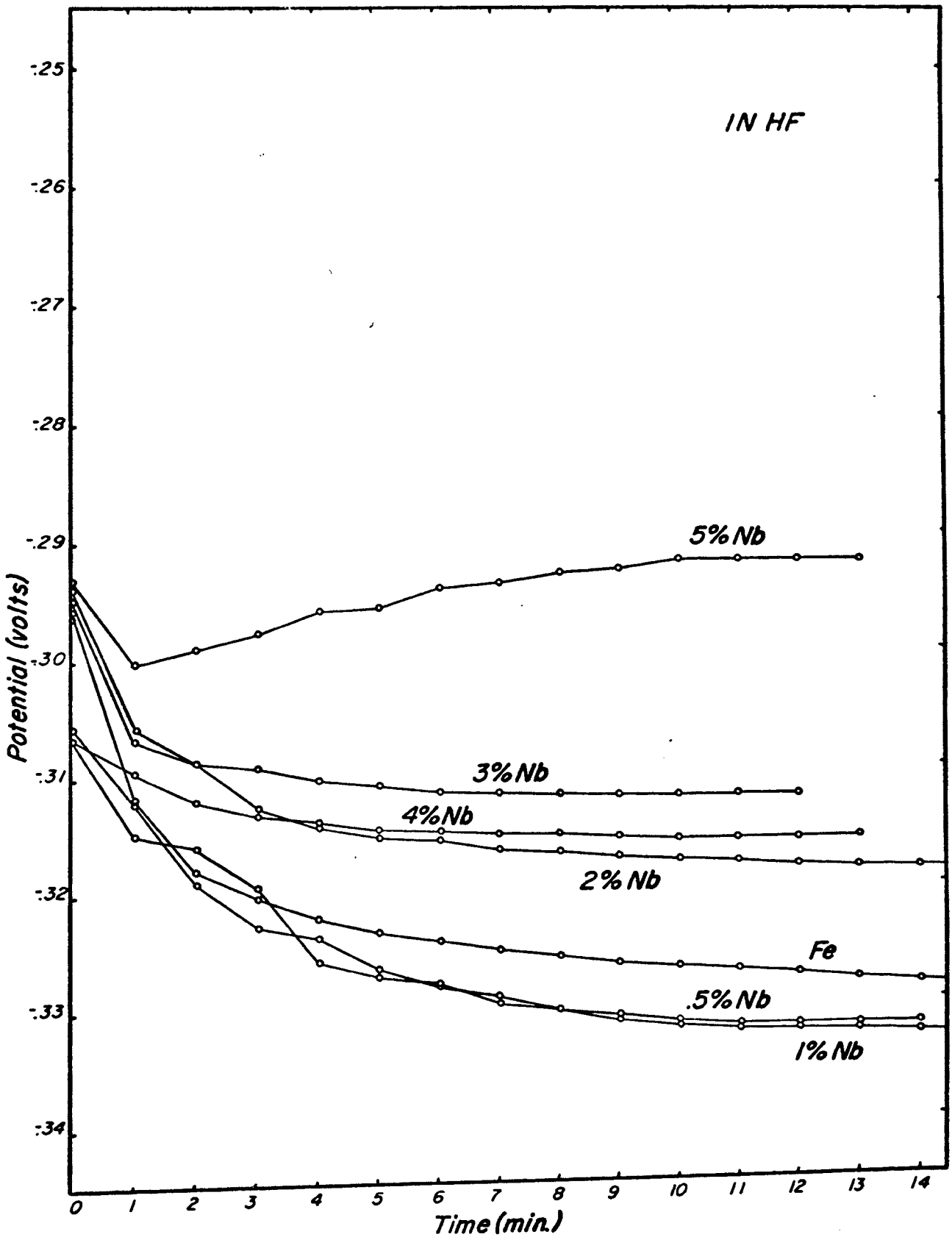

FIGURE 8

POTENTIAL VS. TIME FOR IRON AND IRON-NIOBIUM ALLOYS IN 1N HF

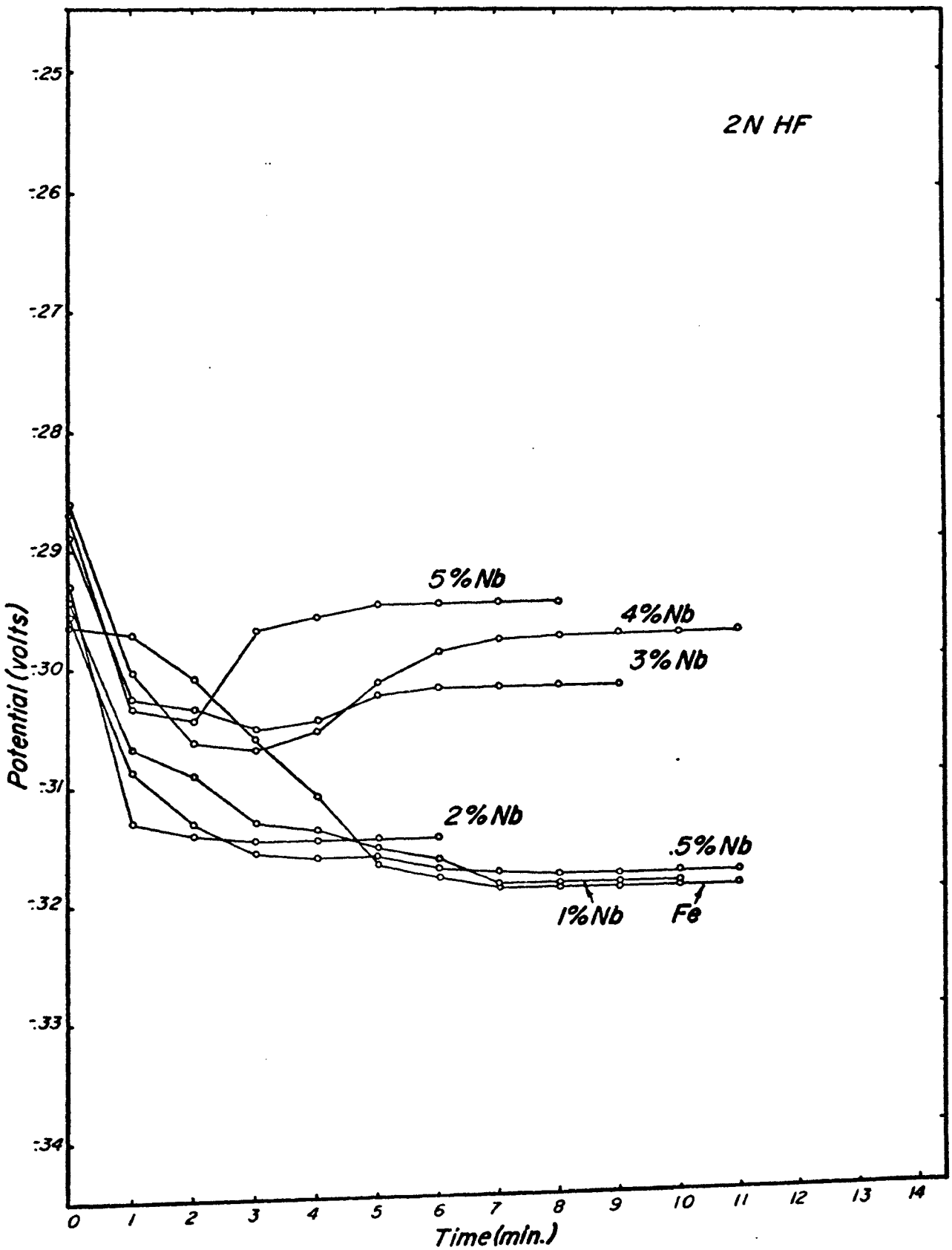

FIGURE 9

POTENTIAL VS. TIME FOR IRON AND IRON-NIOBIUM ALLOYS IN 2N HF

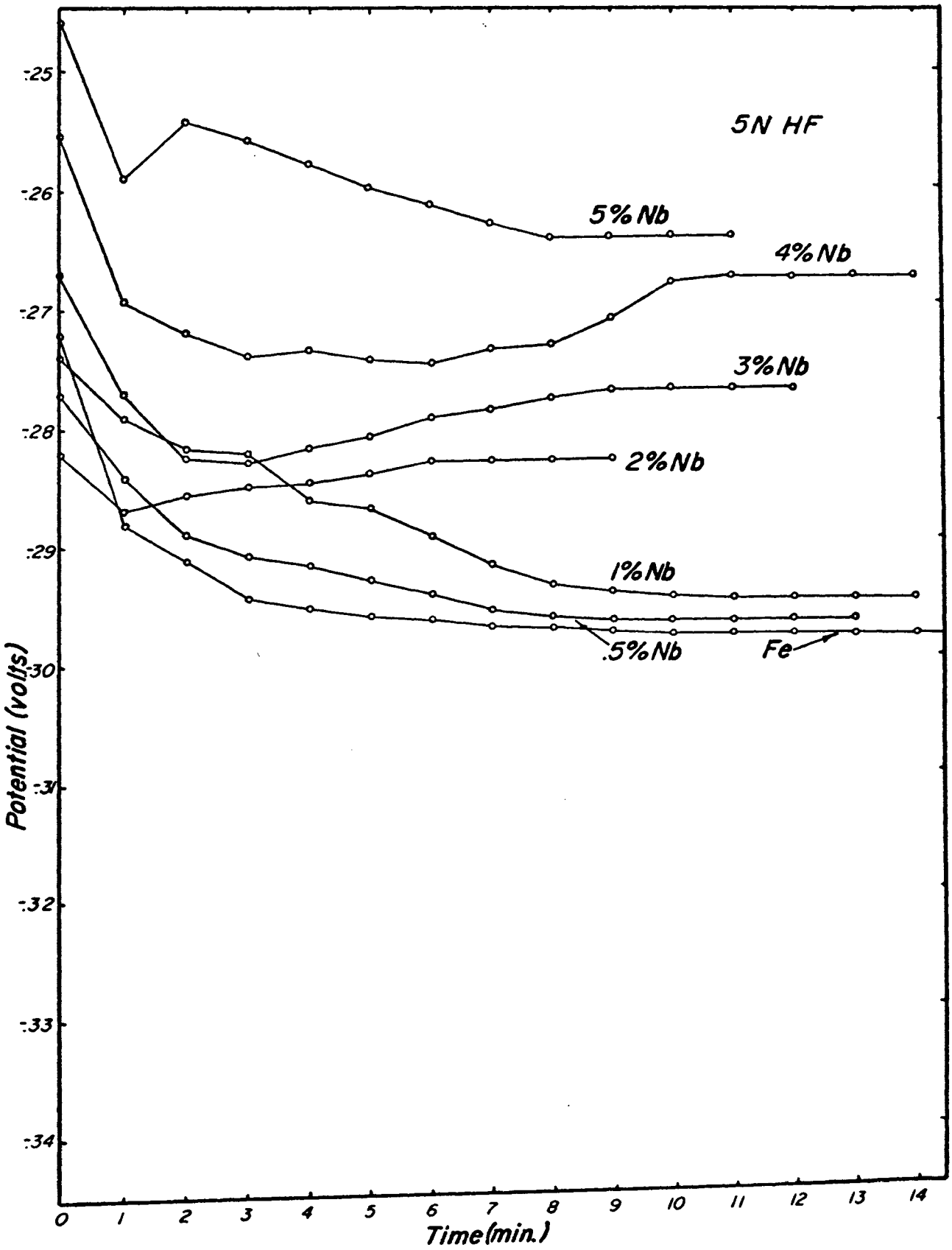

FIGURE 10

POTENTIAL VS. TIME FOR IRON AND IRON-NIOBIUM ALLOYS IN 5N HF

TABLE 80

THE ELECTROCHEMICAL POTENTIAL OF

Nb IN .05N HF

TIME (min.)	POTENTIAL (volts)
0	-.1492
1	-.1525
2	-.1600
3	-.1675
4	-.1716
5	-.1762
6	-.1799
7	-.1820
8	-.1834
9	-.1844
10	-.1850
11	-.1851
12	-.1851
13	-.1851
14	-.1851

TABLE 81

THE ELECTROCHEMICAL POTENTIAL OF Nb IN .1N HF

TIME (min.)	POTENTIAL (volts)
0	-.1875
1	-.1942
2	-.1994
3	-.2030
4	-.2051
5	-.2080
6	-.2108
7	-.2137
8	-.2163
9	-.2175
10	-.2187
11	-.2190
12	-.2198
13	-.2199
14	-.2200
15	-.2200
16	-.2200
17	-.2200

TABLE 82

THE ELECTROCHEMICAL POTENTIAL OF Nb IN 1N HF

TIME (min.)	POTENTIAL (volts)
0	-.2842
1	-.2929
2	-.2958
3	-.2952
4	-.2950
5	-.2951
6	-.2950
7	-.2937
8	-.2937
9	-.2937
10	-.2937

TABLE 83

THE ELECTROCHEMICAL POTENTIAL OF Nb IN 3N HF

TIME (min.)	POTENTIAL (volts)
0	-.3009
1	-.3088
2	-.3082
3	-.3062
4	-.3048
5	-.3038
6	-.3035
7	-.3028
8	-.3025
9	-.3019
10	-.3015
11	-.3015
12	-.3030
13	-.3025
14	-.3025
15	-.3025
16	-.3025

TABLE 84

THE ELECTROCHEMICAL POTENTIAL OF

Nb IN 6N HF

TIME (min.)	POTENTIAL (volts)
0	-.2970
1	-.3035
2	-.3035
3	-.3032
4	-.3032
5	-.3030
6	-.3030
7	-.3029
8	-.3028
9	-.3025
10	-.3025
11	-.3025
12	-.3025

TABLE 85

THE ELECTROCHEMICAL POTENTIAL OF

NbH_x IN .1N HF

TIME (min.)	POTENTIAL (volts)
0	-.1769
1	-.2010
2	-.2044
3	-.2060
4	-.2062
5	-.2080
6	-.2079
7	-.2079
8	-.2079
9	-.2079

TABLE 86

THE ELECTROCHEMICAL POTENTIAL OF

NbH_x IN 1N HF

TIME (min.)	POTENTIAL (volts)
0	-.2260
1	-.2345
2	-.2339
3	-.2330
4	-.2323
5	-.2325
6	-.2330
7	-.2335
8	-.2336
9	-.2336
10	-.2336
11	-.2336

TABLE 87

THE ELECTROCHEMICAL POTENTIAL OF NbH_x IN 6N HF

TIME (min.)	POTENTIAL (volts)
0	-.2190
1	-.2525
2	-.2510
3	-.2506
4	-.2507
5	-.2505
6	-.2500
7	-.2500
8	-.2499
9	-.2495
10	-.2495
11	-.2495
12	-.2495

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VITA

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